

UNIVERSITÀ DEGLI STUDI DI PADOVA

DIPARTIMENTO DI TECNICA E GESTIONE DEI SISTEMI INDUSTRIALI
CORSO DI LAUREA IN INGEGNERIA GESTIONALE

TESI DI LAUREA MAGISTRALE

**Analysis of microstructure and thermal
properties of coated and uncoated
Al-Si alloys**

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2022/2023

ABSTRACT

The aim of this project is to study the thermal properties of aluminium-silicon samples. In order to do that a microstructural analysis is carried out first. Using a spectrometer, the alloy found is an EN 46000, thus an alloy with 9% of silicon and 3% of copper. Using the microscope, the compounds of the samples are analyzed, three different ones are found: Fe- α , AlCu₂ and compounds containing both Cu and Fe. Knowing the microstructure of the specimens, their thermal properties are analysed: thermal conductivity, thermal diffusivity, specific heat and thermal effusivity. To do this, the Hot Disk methodology is used; it is a technology that allows several rapid, nondestructive tests. Thanks to this machine these properties are calculated using a sensor placed between two specimens of the same material. To start the measurement there are two parameters required: output power and measuring time. After defining these inputs, there are four parameters in particular that must be considered when obtaining results. Once a suitable combination of power and time has been found, which gives results within the ranges of the parameters, the results can then be analysed. The method used is the slab method, characterised by radially propagation of heat into the sample. Three different pairs of samples are tested: one with a polished and an unpolished side and two with a coated and an uncoated (unpolished) side. There are two different types of coating: one is thinner, it is only 15-20 μm and the other one is 130-150 μm . Comparing the values of the properties obtained with the ones obtained 10 years ago the thermal properties trend as power increases remains the same for an uncoated sample. The study of this type of alloy is crucial for the future, as it is a material that will be used extensively from both primary production and waste. The possibility that by using suitable coatings its thermal properties will improve, it is not to be underestimated.

1. LETTERATURA

L'alluminio, *Al* nella tavola periodica, è uno dei materiali più diffusi e usati quotidianamente sulla terra soprattutto nelle applicazioni in ambito tecnologico. Esso è infatti duttile e ha delle ottime proprietà termiche e elettriche grazie alla sua struttura cubica a facce centrate; tuttavia non ha un'elevata resistenza alla rottura. L'alluminio solitamente viene utilizzato legato ad altri elementi, come il *silicio*. Viene utilizzato molto in fonderia, in quanto rende la lega molto fluida e di conseguenza va a diminuire la probabilità di ritiro da solidificazione.

In questa tesi su dei campioni di AlSi si è fatta sia l'analisi microstrutturale sia termica. Per quanto riguarda quest'ultima, è fondamentale definire quattro proprietà che influenzano come il materiale riesce a trasferire il calore e come reagisce ai cambiamenti di temperatura. Le proprietà sono:

1. Conduttività termica: la quantità di energia (calore) che viene scambiata durante un processo attraverso una superficie. Per l'alluminio e le sue leghe, i valori possono variare da 70 a 230 Wm K, a seconda degli elementi alliganti presenti e del loro rapporto quantitativo.
2. Diffusività termica: la propagazione del calore in condizioni non stazionarie, cioè variabili nel tempo. Questa proprietà è intrinseca al materiale. Il valore di riferimento, posseduto dall'alluminio puro, è solitamente 97 mm²s, che tende a diminuire quando si aggiungono elementi per creare leghe.
3. Calore specifico: la quantità di energia necessaria per modificarne la temperatura. La sua definizione è: "*La capacità termica di un corpo o di un sistema è la quantità di calore necessaria per aumentare di 1°C la temperatura di un'unità di massa*".
4. Effusività termica: la capacità di un materiale di scambiare calore con l'ambiente circostante. Dipende da una combinazione dei parametri spiegati in precedenza come la conduttività termica e la capacità termica specifica.

2. ANALISI MICROSTRUTTURALE

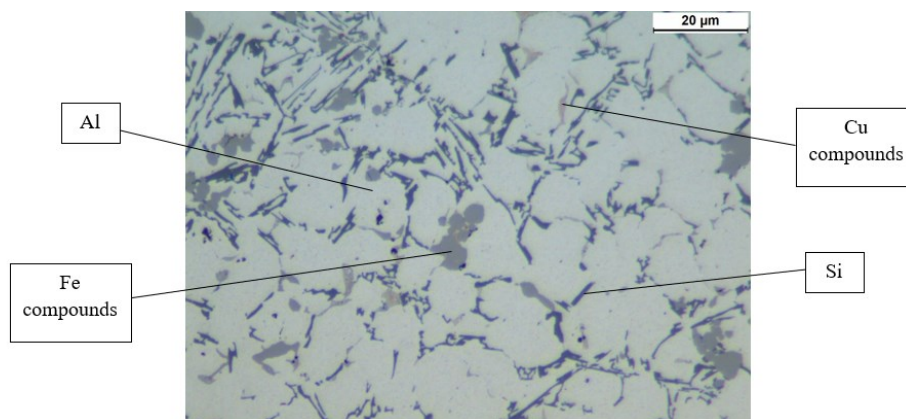
Inizialmente è stata definita la composizione del materiale grazie allo spettrometro di massa. I risultati ottenuti sono riportati nella tabella di seguito.

<i>Elementi</i>	<i>Valori ottenuti</i>	<i>Valori lega EN 46000</i>
Al	84,82	Rimanente
Si	9,37	8,0 - 11,0
Fe	0,87	0,6 - 1,1
Cu	3,31	2,0 - 4,0
Mn	0,24	0,55
Mg	0,19	0,15 - 0,55
Zn	1,03	1,2
Cr	0,03	0,15
Ni	0,05	0,55
Ti	0,04	0,20
Pb	0,08	0,35
Sn	0,01	0,25

I valori trovati rientrano nell'intervallo dei valori standard di una lega EN 46000 (lega con il 9% di Si e il 3% di Cu).

La microstruttura del campione è stata analizzata con due microscopi, quello ottico e lo Scanning Electron Microscope (SEM). Il primo genera le immagini grazie alla luce riflessa dalle lenti, mentre il SEM genera l'immagine grazie a un fascio di elettroni che viene proiettato sul materiale in una camera a vuoto.

Dall'analisi è emerso che il materiale è formato da una matrice di alluminio, dall'eutettico lamellare di silicio e da due principali composti: uno di ferro e l'altro di rame come si può notare nella figura sottostante.



Grazie all'analisi al SEM si è riusciti a definire l'entità di questi composti: Fe- α , la cui formula è $Al_{15} Si_2 (Fe, Mn, Cr, Cu)_3$, $AlCu_2$ e una fase intermedia tra le due precedenti in cui ci sono composti contenenti sia Cu che Fe. Quest'ultima è dovuta probabilmente alla presenza di composti $Al_{15} Si_2 (Fe, Cu)_3$ la cui composizione chimica è molto simile a quella del Fe- α .

L'analisi con lo spettrometro ha rivelato una grande quantità di zinco. Gli studi effettuati mostrano che lo zinco è presente nella matrice, nei composti con il rame e nei composti della fase intermedia.

Come ultima prova meccanica, è stata effettuata la prova di durezza. La durezza di un materiale è definita come la sua resistenza alla penetrazione da parte di un materiale più duro. Per questo campione è stata utilizzata la prova di durezza Brinell, che utilizza una sfera di acciaio temprato con un diametro di 2,5 mm come penetratore. Il carico utilizzato è di 62,5 kg. Viene applicato per circa 30 s, dando alla lega il tempo di deformarsi. Il valore della durezza del campione ottenuta è:

$$HB_{2,5/62,5} = 90$$

3. SOFTWARE E STRUMENTI PER L'ANALISI TERMICA

La strumentazione utilizzata per misurare le proprietà termiche dei campioni fa parte della tecnologia Hot Disk, la quale permette di effettuare numerosi test rapidi e non distruttivi. Le misurazioni vengono effettuate utilizzando un sensore posto in uno specifico supporto, in modo che si trovi al centro dello strumento. Successivamente si prendono i campioni: una metà del campione viene posta sopra il sensore e l'altra sotto in modo che il sensore rimanga ortogonale e in mezzo ai campioni. Solitamente viene utilizzato un pezzo di acciaio sopra ai campioni, in questo modo quando si chiude la vite che serve a fermarli, non vengono danneggiati, inoltre garantisce che la pressione di serraggio sia unidirezionale. I pezzi devono essere fissati saldamente tra loro per evitare la presenza d'aria nel sistema, che potrebbe surriscaldare lo strumento e danneggiare così il sensore. Il tutto viene chiuso sotto un coperchio cilindrico. Il luogo in cui vengono effettuate le misurazioni deve essere un ambiente stabile, isotermico, privo di vibrazioni e con umidità costante.



Il software utilizzato richiede due dati di input:

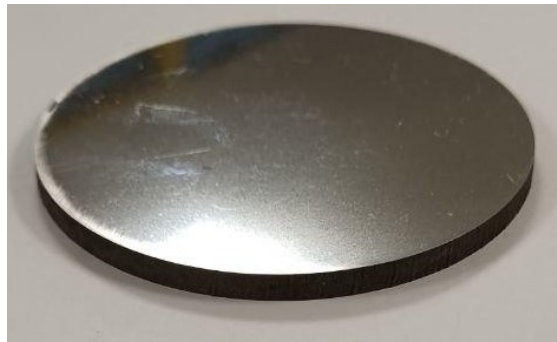
- *Output Power*: L'energia elettrica utilizzata per riscaldare il sensore durante l'esperimento.
- *Measuring Time*: Il tempo di misurazione, che descrive il tempo impiegato dalla macchina per ottenere i dati. L'obiettivo è quello di trovare la combinazione giusta tra *output power* e *measuring time* per ogni coppia di campioni. Quando inizia la misurazione, la temperatura nel sistema aumenta grazie

alla corrente che attraversa le spirali. La scelta del sensore è molto importante, ce ne sono di diversi tipi e dimensioni in base alla forma e al materiale del campione da testare. In questa tesi è stato usato un sensore rotondo con le spirali di Nichel.

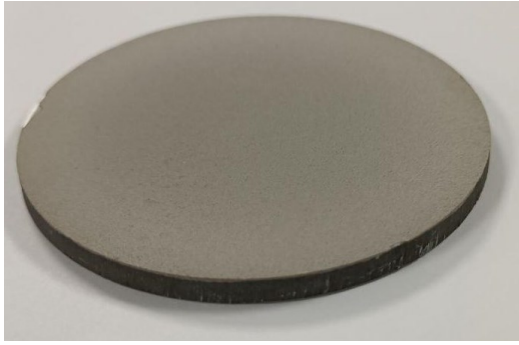
I campioni studiati hanno una forma cilindrica con un diametro di 7,5 cm e un'altezza di 4,5 mm. Sono state testate tre diverse coppie di campioni: una con un lato lucidato e uno non lucidato e due con un lato rivestito e uno non rivestito (e non lucidato). Esistono due diversi tipi di rivestimento: uno più sottile, di soli 15-20 μm , e l'altro di 130-150 μm . Il rivestimento è realizzato in ossido di polietilene (PEO), un materiale termoplastico solubile in acqua.



Superficie non lucida



Superficie lucida



Superficie rivestita (15-20 μm)



Superficie rivestita (130 – 150 μm)

Utilizzando questo macchinario ci sono vari metodi che si possono utilizzare che misurano le diverse proprietà in modi diversi. In particolare ne sono stati utilizzati:

- *Isotropic*: calcola le proprietà termiche di diversi campioni (solidi, in polvere, liquidi) ottenendo dei valori che sono un valore medio del volume del campione.
- *Slab*: i campioni scelti per questo modulo sono metalli, semiconduttori e grafite. I dati di ingresso sono lo spessore del campione, il tempo di misura e la potenza di uscita. Per questi motivi è sempre conveniente utilizzare due metà del campione per le misure.
- *Thin film*: si utilizza nel caso di isolanti sottili, nell'intervallo da 20 a 500 μm . Come per il metodo a lastre, sono necessarie due metà.

Per ogni metodo, per capire se i risultati sono soddisfacenti e la combinazione tra *output power* e *measuring time* risulta adeguata, vengono analizzati 4 parametri:

1. *Probing depth* - la quantità di calore che si propaga nei campioni.
2. *Temperature Increase* - la temperatura tra il primo punto selezionato e l'ultimo. Di solito la temperatura deve crescere di almeno 0,05 K.
3. *Ratio between Total and the Temperature Increase* - l'incremento di temperatura totale, quindi corrisponde al valore di temperatura dell'ultimo campione. Dipende dalle misurazioni, ma in genere questo rapporto deve essere superiore a 1,25 K.
4. *Ratio between Total Time for the measurement and the Characteristic Time* - Ha un intervallo compreso tra 0,33 e 1.

Inizialmente viene utilizzato il metodo *Isotropic* per i campioni con una superficie lucidata e l'altra non lucidata. In letteratura i valori della conduttività termica delle

leghe di alluminio variano dai 120 ai 160 W/mK. In questo caso invece i valori medi ottenuti sono circa 42 W/mK per la superficie lucidata e 43 W/mK per quella non lucidata. Successivamente, viene utilizzato il metodo *Slab* caratterizzato dalla propagazione radiale del calore nel campione. Per questo motivo per quanto riguarda il parametro *Probing Depth* il limite è leggermente inferiore al diametro dei campioni, per questo motivo il valore massimo di questo parametro è impostato a 35 mm. Per i campioni ricoperti è stato utilizzato il metodo *Thin Film*, poiché indicato per questo tipo di materiali. Viene, infatti, utilizzato per valutare la conducibilità termica di pellicole o rivestimenti isolanti sottili. Il processo di misurazione prevede il calcolo della resistenza termica sul film, che consente poi di determinare la conduttività termica tenendo conto dello spessore dello stesso. Visti i risultati ottenuti utilizzando i diversi approcci, si è deciso quindi di utilizzare il metodo *Slab* per tutti i campioni.

4. RISULTATI E CONFRONTI

Il primo campione studiato è quello non ricoperto. Nella tabella sottostante sono riportati i valori trovati delle tre proprietà termiche. Il *measuring time* utilizzato è stato per tutte le prove pari a 1s.

Proprietà termiche del campione non ricoperto

	<i>Output Power</i> [W]	<i>Conduttività term.</i> [W/mK]	<i>Diffusività term.</i> [mm ² /s]	<i>Calore specifico</i> [MJ/m ³ K]
<i>Lato lucido</i>	1	154,06	43,16	3,57
	1,2	159,59	47,95	3,33
	1,5	159,59	50,42	3,17
<i>Lato non lucido</i>	0,8	161,97	52,03	3,11
	1	168,02	42,62	3,94
	1,1	168,86	50,21	3,75
	1,2	168,93	42,99	3,93
	1,3	165,44	40,53	4,08
	1,5	166,51	45,88	3,63

Il secondo campione studiato è quello ricoperto con il rivestimento più sottile (15-20 µm). Come per il campione precedente, per confrontare i valori delle proprietà termiche ottenute, il *measuring time* utilizzato è pari a 1 s.

Proprietà termiche del campione rivestito (15-20 μm)

	Output Power [W]	Conduttività term. [W/mK]	Diffusività term. [mm ² /s]	Calore specifico [MJ/m ³ K]
Lato rivestito	0,8	189,90	92,39	2,06
	0,9	179,18	85,35	2,23
	1	180,50	85,91	2,10
Lato non rivestito	0,8	177,79	62,84	2,83
	1	179,04	74,12	2,42
	1,2	168,47	51,01	3,30
	1,5	169,60	44,75	3,79

Per quanto riguarda il terzo campione, quello ricoperto con il rivestimento più spesso (130-150 μm), vengono effettuate diverse misure. L'intervallo del *measuring time* per il lato rivestito varia da 2 a 5 s, mentre per il lato non rivestito da 1 a 5 s. Il range invece dell'*output power* è da 0,70 a 0,75 W, mentre per il lato non rivestito da 0,8 a 1,0 W. Visti i valori di input diversi, per questo campione, le due superfici vengono analizzate separatamente. Viene studiato prima il lato rivestito. La tabella sottostante riporta i valori delle proprietà utilizzando un tempo di misura pari a 5 s.

Proprietà termiche del lato rivestito del campione rivestito (130-150 μm)

	Output Power [W]	Conduttività term. [W/mK]	Diffusività term. [mm ² /s]	Calore specifico [MJ/m ³ K]
Lato rivestito	0,7	140,46	51,09	2,75
	0,75	142,17	56,92	2,50
Valore medio		141,31		

I dati riportati nella tabella evidenziano che i valori delle proprietà termiche di questo lato del campione hanno valori inferiori rispetto a quelli dei campioni precedenti. Questa situazione può essere paragonata a quella di due resistori in parallelo: uno rappresentato dal rivestimento PEO e l'altro dalla base in AlSi. Il sistema può essere visto, per analogia, come un collegamento in parallelo di due resistenze in cui la corrente che circola nei due rami del circuito è sostituita dalla potenza termica. In questo caso, quindi, il calore generato dal passaggio della corrente attraverso il sensore viene suddiviso tra le due parti del campione.

Di conseguenza i valori della conduttività termica seguono questa regola:

$$k_{\Sigma} \cdot h_{\Sigma} = k_{coat} \cdot h_{coat} + k_{base} \cdot h_{base} \cdot x$$

in which x is:

$$x = \frac{h_{base\ eff.}}{h_{base}}$$

per questo motivo i valori sono minori.

Per quanto riguarda il lato non rivestito di questo campione non è stata trovata una combinazione adeguata tra *measuring time* e *output power*.

Il campione non rivestito è stato studiato anche 10 anni fa con un diverso macchinario, ma della stessa tipologia. Tuttavia, il metodo TPS e il materiale del sensore sono gli stessi: le proprietà sono state rilevate in direzione radiale utilizzando il metodo *Slab*. I risultati ottenuti sono riportati nella tabella sottostante.

Proprietà termiche del campione non rivestito (10 anni fa)

	<i>Conduttività term.</i> [W/mK]	<i>Diffusività term.</i> [mm ² /s]	<i>Calore specifico</i> [MJ/m ³ K]	<i>Conduttività term.</i> [W/mK]
<i>Lato non rivestito</i>	4,5	142,7	56,0	2,551
	5	143,2	56,2	2,549
	5,5	139,5	55,8	2,500

Questi risultati sono leggermente diversi da quelli ottenuti ora. Molto probabilmente lo sviluppo e l'aggiornamento di nuovi macchinari o tecnologie diverse, ha modificato le prestazioni e le caratteristiche dell'apparecchiatura influenzando l'accuratezza e la precisione delle misure. Tuttavia, la somiglianza nell'andamento dei dati è evidente, in quanto suggerisce che, nonostante le differenze nelle apparecchiature, la relazione fondamentale tra *output power* e i valori ottenuti è coerente: come dieci anni fa, anche oggi la conduttività termica e la diffusività termica sono indipendenti dalla potenza.

5. CONCLUSIONI

Lo scopo di questo progetto di tesi è stato lo studio delle proprietà termiche dei campioni in lega di alluminio. Lo studio della microstruttura del campione con lo spettrometro e i microscopi mostra che la lega di questo progetto finale è una EN 46000, una lega di alluminio con il 9% di silicio, che ha numerose applicazioni in vari ambiti industriali. Le proprietà analizzate sono la conducibilità termica, la diffusività termica e il calore specifico. Le proprietà sono state studiate con la macchina Hot Disk e il metodo TPS.

Dall'analisi dei tre campioni, si evince che:

- Per il lato non rivestito dei campioni sono stati provati due metodi: il metodo *Isotropic*, ottenendo dei valori troppo bassi rispetto a quanto previsto, e il metodo *Slab*, in cui il calore si propaga radialmente attraverso il campione. Quest'ultimo è il metodo usato per determinare le proprietà termiche.
- Anche per il lato ricoperto sono stati testati due metodi: il metodo *Thin Film*, che sarebbe stato adatto per il calcolo delle proprietà di strati sottili, senza ottenere combinazioni adeguate di potenza-tempo, e il metodo *Slab*, che ha restituito valori di proprietà simili a quelli attesi.

In conclusione, il metodo migliore per analizzare questi campioni è stato il metodo *Slab*. I risultati sono riportati nella tabella seguente.

Risultati ottenuti dai campioni in esame

<i>Slab Method</i>	<i>Output power [W]; Meas. time [s]</i>	<i>Conduttività term. [W/mK]</i>	<i>Diffusività term. [mm²/s]</i>	<i>Calore specifico [MJ/m³K]</i>
Lato lucido campione non ricoperto	1 - 1,5; 1	157,75	47,177	3,35
Lato non lucido campione non ricoperto	0,8 - 1,5; 1	166,42	50,506	3,42
Lato rivestito campione rivestito (130-150µm)	0,7 - 0,75; 5	142,59	72,144	2,08
Lato non rivestito campione rivestito (15-20µm)	0,8 - 1,5; 1	172,84	53,164	3,38
Lato rivestito campione rivestito (15-20µm)	0,75 - 1; 1	177,48	72,05	2,55

Non è stato possibile calcolare i parametri per la superficie non rivestita del campione con copertura di 130-150 µm. Infatti, in base ai punti scelti nel diagramma transitorio e al tempo di misurazione utilizzato, i risultati delle tre proprietà mostrano che: utilizzando un tempo ridotto, solo i valori della conduttività termica rientravano nel range atteso e, utilizzando un tempo leggermente superiore, la conduttività termica diminuiva.

Confrontando questi dati con quelli ottenuti 10 anni fa, sebbene la strumentazione fosse diversa, ma il metodo per ricavare i valori utilizzato fosse lo stesso (*Slab Method*), si nota come l'andamento delle proprietà termiche all'aumentare della potenza rimane lo stesso per il campione non rivestito. Lo studio di questo tipo di lega è fondamentale per il futuro, in quanto si tratta di un materiale che verrà ampiamente utilizzato sia ottenuto dalla produzione primaria, che dagli scarti. La possibilità che utilizzando rivestimenti adeguati le sue proprietà termiche migliorino, non è da sottovalutare. Il metodo TPS (Transient Plane Source) è anche molto innovativo, in quanto molto preciso, non degrada i campioni e permette di ottenere molti valori in poco tempo. D'altra parte, trovare le giuste combinazioni di potenza-tempo e il giusto metodo con cui studiare il materiale non è così immediato.

TABLE OF CONTENTS

LIST OF FIGURES	III
LIST OF TABLES	VII
INTRODUCTION	1
1. LITERATURE SURVEY	5
1.1 Aluminium	5
1.2 Aluminium-silicon alloys.....	9
1.3 Thermal properties	12
1.3.1 Thermal conductivity	14
1.3.2 Thermal diffusivity.....	15
1.3.3 Heat capacity	16
1.3.4 Thermal effusivity	16
2. MICROSTRUCTURE	19
2.1 Chemical composition.....	19
3.2 Microstructure	20
3.3 Hardness test	28
3. TOOLS AND METHODS	31
3.1 Hot Disk	31
3.2 Transient Plane Source.....	39
3.3 How to measure.....	40
4. EXPERIMENTAL WORK	47
4.1 Test.....	48
4.1.1 Isotropic Method	48
4.1.2 Slab Method for uncoated side of specimens.....	49
4.1.3 Thin Film Method for coated side of specimens.....	58
4.1.4 Slab Method for coated side of specimens.....	63
4.2 Parameters	67
5. DATA ANALYSIS	69
5.1 Uncoated sample	71
5.2 Coated sample (15-20 μm).....	73
5.2 Coated sample (130-150 μm).....	75
5.5 Comparison between found data and old data	81
5.6 Comparison between found data and literature data	84
CONCLUSIONS	85
BIBLIOGRAPHY	87

LIST OF FIGURES

Figure 1 - Production of Aluminium in the last 22 years	1
Figure 1.1 - Bayer process	6
Figure 1.2 - Hall-Heroult cell	7
Figure 1.3 - Al-Si Phase Diagram	11
Figure 1.4 - Example of a solid	13
Figure 2.1 - Tested sample	19
Figure 2.2 - Sample ready for the analysis (the red part is the one near to the mould)	21
Figure 2.3 - Up: full cross-section (red line is the side next to the mould), Down: zoom on oxide defects on the surface	21
Figure 2.4 - Frame next to the mould, on the left magnitude 100x, on the right magnitude 200x	22
Figure 2.5 - Frame in the central part of the sample, on the left magnitude 100x, on the right magnitude 200x	22
Figure 2.6 - Material constituents	23
Figure 2.7 - SEM: on the left magnitude 1000x, on the right magnitude 4000x .	23
Figure 2.8 - SEM1: on the left magnitude 4000x, on the right the spectrum of the components of the circle	24
Figure 2.9 - SEM2: on the left magnitude 4000x, on the right the spectrum of the components of the red circle	25
Figure 2.10 - SEM3: on the left magnitude 4000x, on the right the spectrum of the components of the red circle	25
Figure 2.11 - White points: on the left magnitude 4000x, on the right the spectrum of the components	26
Figure 2.12 - Sample after chemical attack (Cu), magnitude 50x (left) and magnitude 250x (right)	27
Figure 2.13 - Sample after chemical attack (Fe), magnitude 50x (left), magnitude 250x (right)	27
Figure 2.14 - Sample after chemical attack (eutectic) (left), magnitude 250x (right)	28

Figure 2.15 - Hardness test	29
Figure 3.1 - TPS2500 S	31
Figure 3.2 - Sensor Kapton C5501	34
Figure 3.3 - Isotropic methods parameters	37
Figure 3.4 - How to position sensor and specimens	40
Figure 3.5 - Drift Chart	42
Figure 3.6 - Transient Chart	42
Figure 3.7 - Calculated Chart	43
Figure 3.8 - Residual Chart	43
Figure 3.9 - Example of warnings	45
Figure 4.1 - Unpolished side	47
Figure 4.2 - Polished side	47
Figure 4.3 - Coated side (15 - 20 μm)	47
Figure 4.4 - Coated side (130 - 150 μm)	47
Figure 4.5 - Transient Chart of the polished side of the uncoated sample with the Slab Method	51
Figure 4.6 - Probing Depth of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)	52
Figure 4.7 - Temperature Increase of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)	52
Figure 4.8 - Total/Temp. Incr. of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)	53
Figure 4.9 - Total/Characteristic Time of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)	53
Figure 4.10 - Residual Chart of the polished side of the uncoated sample with the Slab Method	54
Figure 4.11 - Parameters of the unpolished side of the uncoated sample with the Slab Method (green columns are acceptable)	55
Figure 4.12 - Parameters of the uncoated side of the coated sample (15 - 20 μm) with the Slab Method (green columns are acceptable)	57
Figure 4.13 - Parameters of the uncoated side of the coated sample (130 - 150 μm) with the Slab Method (green columns are acceptable)	58

Figure 4.14 - Parameters of the coated side of the coated sample (15 - 20 μm) with the Thin Film Method (green columns are acceptable)	60
Figure 4.15 - Parameters of the coated side of the coated sample (130 - 150 μm) with the Thin Film Method (green columns are acceptable)	62
Figure 4.16 - Parameters of the coated side of the coated sample (15 - 20 μm) with the Slab Method (green columns are acceptable)	64
Figure 4.17 - Thermal Diffusivity of the coated side of the coated sample (15-20 μm) with the Slab Method	65
Figure 4.18 - Parameter of the coated side of the coated sample (130 - 150 μm) with the Slab Method (green columns are acceptable)	66
Figure 5.1 - Single resistor	70
Figure 5.2 - Parallel connection of two resistors	70
Figure 5.3 - Thermal Conductivity and Thermal Diffusivity of the uncoated sample	72
Figure 5.4 - Specific Heat of the uncoated sample	72
Figure 5.5 - Comparison of Thermal Conductivity of coated and uncoated sample	74
Figure 5.6 - Thermal Conductivity and Thermal Diffusivity of the coated sample (15-20 μm)	74
Figure 5.7 - Specific Heat of the coated sample (15-20 μm)	75
Figure 5.8 - Comparison of thermal conductivities	76
Figure 5.9 - Thermal properties of the coated side of the coated sample (130-150 μm)	78
Figure 5.10 - Comparison of Thermal Diffusivity of the uncoated side of the coated sample (130-150 μm) with the uncoated sample	79
Figure 5.11 - Comparison of Specific Heat of the uncoated side of the coated sample (130-150 μm) with the uncoated sample	79
Figure 5.12 - Thermal Conductivity and Thermal Diffusivity of the uncoated sample studied today and 10 years ago	83

LIST OF TABLES

Table 1.1 - General characteristic of Aluminium	5
Table 2.1 - Chemical composition of the sample	19
Table 2.2 - Chemical composition of the first compound found	24
Table 2.3 - Chemical composition of the second compound found	25
Table 2.4 - Chemical composition of the third compound found	26
Table 2.5 - Hardness test	29
Table 3.1 - Characteristics of TPS2500 S	32
Table 4.1 - Parameters of the Isotropic Method	48
Table 4.2 - Output power/measuring time combinations for the polished side of the uncoated sample with the Isotropic Method	49
Table 4.3 - Thermal properties with the Isotropic Method for the sample uncoated	49
Table 4.4 - Parameters of the Slab Method	50
Table 4.5 - Output power/measuring time combinations for the polished side of the uncoated sample with the Slab Method	50
Table 4.6 - Output power/measuring time combinations for the unpolished side of the uncoated sample with the Slab Method	55
Table 4.7 - Output power/measuring time combinations for the uncoated side of the coated sample (15-20 μm) with the Slab Method	56
Table 4.8 - Output power/measuring time combinations for the uncoated side of the coated sample (130-150 μm) with the Slab Method	57
Table 4.9 - Parameters of the Thin Film Method	59
Table 4.10 - Output power/measuring time combinations for the coated side of the coated sample (15-20 μm) with the Thin Film Method	60
Table 4.11 - Thermal properties or the coated side of the coated sample (15-20 μm) with the Thin Film Method	61
Table 4.12 - Output power/measuring time combinations for the coated side of the coated sample (130-150 μm) with the Thin Film Method	62
Table 4.13 - Output power/measuring time combinations for the coated side of the coated sample (15-20 μm) with the Slab Method	63

Table 4.14 - Output power/measuring time combinations for the coated side of the coated sample (130-150 μm) with the Slab Method	65
Table 4.15 - Summary of satisfactory parameters	67
Table 5.1 - Thermal properties of the uncoated sample	72
Table 5.2 - Thermal properties of the uncoated sample	73
Table 5.3 - Thermal properties of the coated side of the coated sample (130-150 μm)	75
Table 5.4 - Changes of k_{coat} as x varies	77
Table 5.5 - Thermal properties of the uncoated side of the coated sample (130-150 μm)	78
Table 5.6 - Thermal properties of the uncoated side of the coated sample (130-150 μm) - Measuring Time = 1s	80
Table 5.7 - Thermal properties of the uncoated side of the coated sample (130-150 μm) - Measuring Time = 5s	80
Table 5.8 - Total/Characteristic Time	80
Table 5.9 - Thermal properties of the uncoated side of the coated sample (130-150 μm) - Measuring Time = 2s	81
Table 5.10 - Thermal properties of the uncoated sample (10 years ago)	81
Table 5.11 - Input: chemical composition	84
Table 6 - Obtained results from the samples under investigation	86

INTRODUCTION

Nowadays, there is a growing demand for materials that can be used in situations that overcome the limits of their mechanical and thermal capabilities. These materials are used in various sectors and it is essential to know and predict how they will perform under various conditions of both mechanical and thermal loads. The behavior of materials is of particular importance in fields such as aerospace, automotive, construction, and manufacturing, where the use of high-performance materials is essential. In particular, the variety of materials considered is wide, it includes polymers, ceramics, fibres, but also metallic materials. The latter are those of most interest for this final project, in particular *Aluminium*. Aluminium occurs naturally in the earth's crust and it is the third most abundant element with oxygen and silicon. It is one of the most widely used materials in the world, its production has more than doubled in the last 20 years (figure 1).

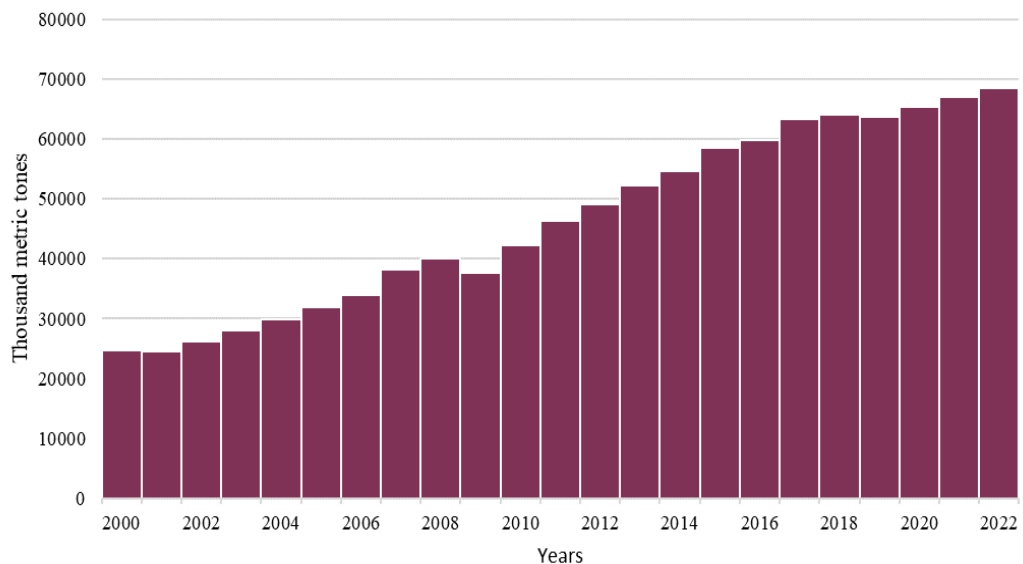


Figure 1 - Production of Aluminium in the last 22 years [1]

Aluminium has a range of desirable properties, such as its lightweight, corrosion resistance, and thermal properties. With the introduction of other materials, aluminium becomes a very versatile and useful material in many applications. The ability to customize the properties of alloys through the introduction of elements offers a wide range of options for a not heavy design, strong and sustainable parts.

Its properties are amplified, so that aluminium becomes an increasingly challenging material that can be used in various fields. Its thermal properties are very important, as the alloys possess a very high thermal conductivity that allows them to exchange heat between one body and another well. These properties change depending on the alloying element. Silicon is one of the most commonly used elements to create alloys with aluminium, as it provides the material with a good fluidity and thus allows it to be better processed in foundry work. These alloys also have good strength, resistance and thermal conductivity, making them useful in a variety of applications, such as automotive engine blocks.

The alloy that is analysed in this final project is an EN 46000, an aluminium alloy containing 9% of silicon and 3% of copper. To find the type of alloy on which to define the thermal properties, a microstructural analysis is initially carried out with a spectrometer and both the light and the scanning electron microscope. After that, the properties of this alloy have been derived using the TPS (Transient Plane Source) method, which is a non-destructive testing technique developed in recent years. The TPS method is known for its speed and precision and does not require repeating the measurements once the results have been obtained. Measurements are made by means of a sensor placed between two samples of the same type. Three pairs of samples are studied in this project. The first pair consists of plain samples with one side polished and the other unpolished. The other two pairs of samples have one side unpolished and the other coated by Polyethylene Oxide, PEO, coating. One pair has a thinner coating (20-15 μm), while the other is thicker (130-150 μm). For each measurement, a combination of measuring time and power is determined. The combinations that provide satisfactory results are selected taking into account other parameters such as probing depth, the temperature increase, the characteristic time and the total temperature. By using various methods, values of thermal properties such as thermal conductivity, specific heat, and thermal diffusivity are obtained.

This final elaborate contains three major parts.

- In the first part the literature survey is introduced. Chapter 2 presents the fundamentals of the history and production of aluminium and provides an overview of various concepts related to its alloys. Additionally, it introduces the four thermal properties that this project focuses on: thermal conductivity, thermal diffusivity, specific heat and thermal effusivity.
- In the second part the type of alloy is found, and the hardness test is carried out.
- Then the Hot Disk machine and the TPS method used are explained.
- The last part is the experimental part. It shows how to find the right combination of output power and measuring time for every side of every sample. This is a critical step in obtaining accurate measurements of the thermal properties of aluminium alloys. Once finding them, the value of the properties are reported. In the end there is a comparison between the data found with the data of the study of ten years ago, to evaluate any differences in the thermal properties of the alloys over time. Then the conclusions are reported.

1. LITERATURE SURVEY

1.1 Aluminium

Aluminium, Al in the periodic table, is one of the most widespread and daily used materials on earth. It is a key material for this period of technological development as more and more applications are possible, as it is ductile and it has excellent thermal and electrical properties due to its face-centered structure; however, it doesn't resist breaking very much. The main characteristics of aluminium are given in the table 1.1.

Table 1.1 - General characteristic of Aluminium [2]

Density at 20°C	2.7 kg/dm ³
Melting temperature	660 °C
Boiling temperature	2450 °C
Thermal conductivity	237 W/mK
Specific heat	93.9 kJ/(kgK)
Latent heat of melting	394.5 kJ/kg
Linear expansion coefficient	24·10 ⁻⁶ K ⁻¹
Young's modulus	70 GPa
Poisson ratio	0.35
Ultimate tensile strength	70 MPa
Yield strength	30 MPa
Vickers hardness	30 HV
Compression strength	30 MPa
Percent elongation	2%

Aluminium is one of the youngest metals for industrial use; its discovery dates back about two centuries when Sir Humphrey Davy hypothesized that aluminium was the salt of a yet unknown metal to which he gave the name "*Aluminium*". Only a few years later a Danish physicist was able to create a few drops of aluminium, by

applying a heat source to a mixture of potassium and aluminium. Thanks to this, one of his followers discovered some of the properties of this material. This discovery caught the attention of many investors who funded the research. The breakthrough year for this material was 1886, when Charles Martin Hall and Paul Heroult simultaneously discovered the electrolytic fusion process to separate oxygen from aluminium starting with alumina [3]. Two years later the Austrian Bayer patented the extraction of aluminium oxide from bauxite. The Bayer and Hall-Heroult processes are still used today to obtain aluminium. Over the years, the development of aluminium was extensive, from the early 1900s it began to be used in the food industry as packaging, and then spread to the medical sector during the Second World War for the production of aerosol cans. The story of aluminium doesn't end here; research and discoveries of new applications are still going on today.

Aluminium can be obtained by two processes:

- Primary route, in which aluminium is extracted from Bauxite
- Secondary route, in this case it is obtained from scrap.

In the primary route, The aluminium is extracted with other elements from bauxite. What is mainly obtained is alumina (Al_2O_3), the methods studied by Bayer can be used to obtain it. The objective of the Bayer cycle then is to obtain alumina from bauxite. The process is synthesized in the following diagram (figure 1.1):

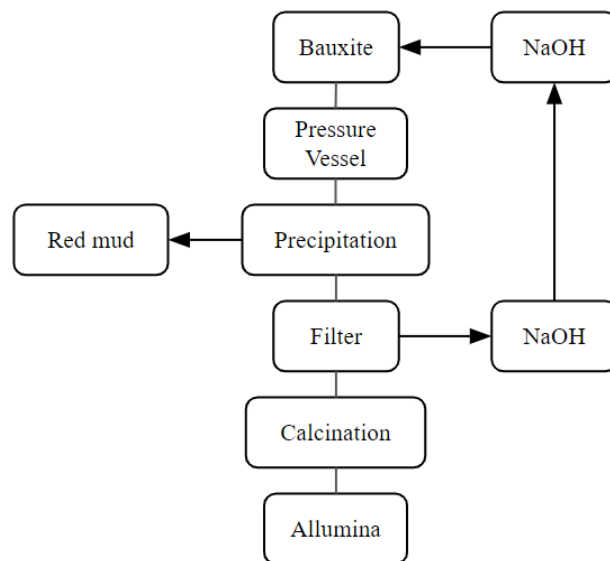


Figure 1.1 - Bayer process

Bayer's cycle begins with the extraction of bauxite, which is a volcanic rock. It has no particular geopolitical problems as deposits can be found all over the world, for example, in Australia, Asia, South America and some deposits in Europe. Initially, the bauxite is mixed with some caustic soda NaOH, inside a pressure vessel that has constant temperature at 240° and pressure at 40 atm. A compound will form within it, containing aluminium, which will float compared to the red mud formed mostly of ferrous materials. Once the sludge is removed, the soda ash goes to separate, which will then be reused in a 90° step. Filtering it out will yield aluminium hydroxide. This will then be calcined at 1300° and in this way alumina will be obtained. Having now obtained the alumina, the next phase is the Hall-Heroult process, in which through an electrolytic process, aluminium is split from oxygen.

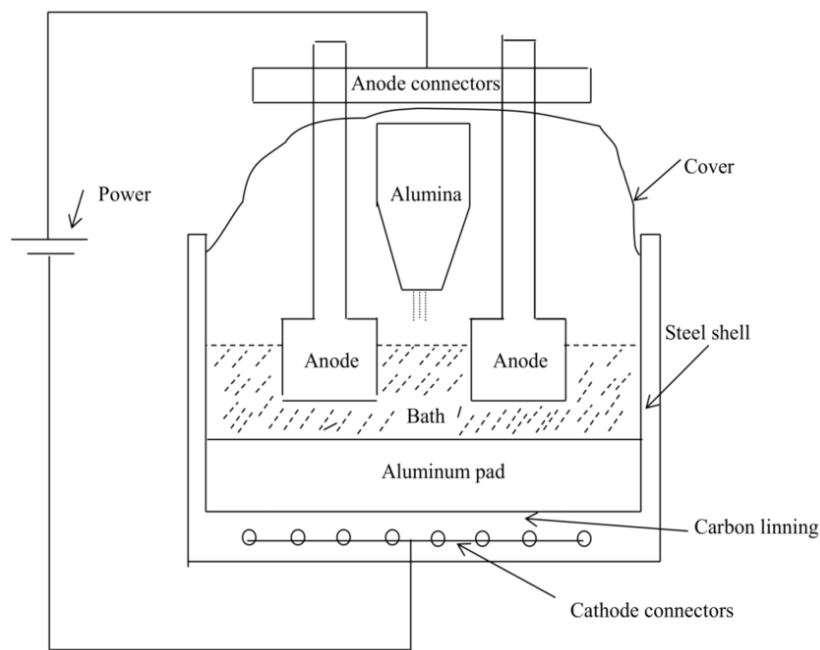


Figure 1.2 - Hall-Heroult cell

In order to do this, liquid alumina is needed, but the temperatures at which it can be found liquid are above 2000°. For this reason, a material is added that when mixed with aluminium forms an eutectic with a lower melting temperature. So inside an electrolytic reduction plant (figure 1.2) alumina is to be placed together with cryolite, a salt based on sodium, aluminium and fluorine. This mixture melts at 950°. Having obtained the liquid a potential difference is applied between two graphite electrodes so that oxygen migrates to the positive pole and aluminium to

the negative pole. Pure aluminium is thus obtained. In quantitative terms, 4 kg of bauxite results in 2 kg of alumina which then results in 1 kg of red mud and 1 kg of water. From this alumina 1 kg of aluminium can be obtained. This type of steel production is becoming increasingly competitive. In fact, in the past 50 years, energy consumption for the production of one kilogram of aluminium has decreased from 20.5 to 13 kWh.

In the secondary route, aluminium can be obtained from two types of scrap:

- New scrap or process scrap, that is the industrial waste during production and fabrication of aluminium products
- Old scrap, meaning aluminium that has already had a concrete application in life and it is being recovered (for example aluminium from cans, windows frames...)

To go about optimizing the scrap, firstly aluminium has to be separated from the constituents that are not of interest. There are different ways to separate materials, the choice depends on which material to be removed. There is the magnetic separation (for example used for iron), the visual separation, gravitational flotation, eddy current system... After separating the different components, the fragmentation phase comes next. In this treatment aluminium is reduced in size and it is separated from other undesirable elements. The next phase is the drying phase, it is used to remove paints and materials covering the scrap. In this phase there is a treatment called *pyrolysis*. In this process the mixed scrap is placed in a furnace, equipped with a steam recovery boiler and a system for the flue gas treatment, because the process is widely used for the conversion of waste materials into energy, fuel, and other valuable products. To remove the paint, the temperature must be around 500°. Then there is the melting transition that occurs at about 900°, which is done with different furnaces depending on the type of scrap. In environmental and energy terms, the secondary production process is more advantageous than the primary one. In fact, the energy consumption to re-melting the scrap is only 5% of the energy to produce the aluminium in the primary route.

So, it can be said that aluminium is used widely because it has several strengths listed below:

- It has infinite availability whether produced by the primary or secondary route
- It is a valuable material in environmental terms, as the energy used to produce it is limited
- It can be 100% recycled countless times, retaining its original characteristics
- It is a lightweight material, weighing approximately one-third of steel
- It resists corrosion, as it is able to oxidise in contact with air, thus creating protection against water and other substances
- It is ductile and malleable, making it easy to work with it at both high and low temperatures
- It can conduct electricity, heat and sound
- It diffuses and reflects light, reducing the dispersion of the brightness and saving energy
- The potential for the future is many; new application areas are already being discovered (for now, the sector in which it is most used is automotive).

With its growing demand, it is crucial to understand the importance of aluminum and its role in our lives. In both cases of production, aluminium is never found in pure form, but always in the form of an alloy. In this way its characteristics can be further extended. The use of aluminium alloys therefore allows a wide range of properties to be customised for specific applications, making them a versatile and valuable material in many sectors.

1.2 Aluminium-silicon alloys

Aluminium alloys are a mixture of aluminium and other elements such as copper, magnesium, silicon, manganese, and zinc, to enhance its properties for example strength, durability, and resistance to corrosion [4, 5, 6, 7, 8, 9]. They are widely used in various industries due to their unique properties. They usually split into two groups based on the processing and treatment they undergo: foundry and plastic deformation. These alloys have a classification set by the *Alluminium Association*. We will only focus on the foundry classification, as the experiments will be carried out on casting alloys. They have an alpha numeric form, with four numbers and one letter. The first number identifies the main element of the alloy, the second and third

values show the specific alloy designations. There may also be a fourth number preceded by a dot to identify the product form: if this is 0 then it defines the castings, if it is 1 or 2 it is the ingots. To define the various versions of an alloy, the numbers are usually preceded by a letter (A, B, C...). Alloys are thus defined as follows:

- 1xx.x, pure aluminum
- 2xx.x, Al - Cu alloys
- 3xx.x, Al - Si - Cu and/or Mg
- 4xx.x, Al - Si
- 5xx.x, Al - Mg
- 7xx.x, Al - Zn
- 8xx.x, Al - Sn
- 9xx.x, Al - other elements
- 6xx.x, unused series

Foundry alloys must be fluid, and when they solidify they must undergo minimal volumetric contraction. In foundry processes it's crucial to study the microstructure of aluminium; in a lot of studies, scientists found out that the finer and more equiaxial the grains are, the better the properties listed below will be:

- Static properties (tensile strength, yield strength, modulus of elasticity...)
- Dynamic properties (thermal conductivity, thermal expansion...)
- Cyclic properties
- The reduction of the hot-tearing phenomenon
- Better distribution of microporosities and intermetallic phases.

To obtain these properties scientists said that by using both nucleating particles and segregating elements in aluminium alloys. One of the elements that is most added to aluminium to form these alloys is silicon. Silicon is an element used extensively in foundry as it makes the alloy much more fluid and consequently goes to decrease the likelihood of solidification shrinkage. Silicon is also used to improve the mechanical properties of the material, however, these alloys can't be heat treated. To go on to better understand aluminium-silicon alloys, let's start with the phase diagram (figure 1.3), that identifies the thermal equilibrium conditions of the material.

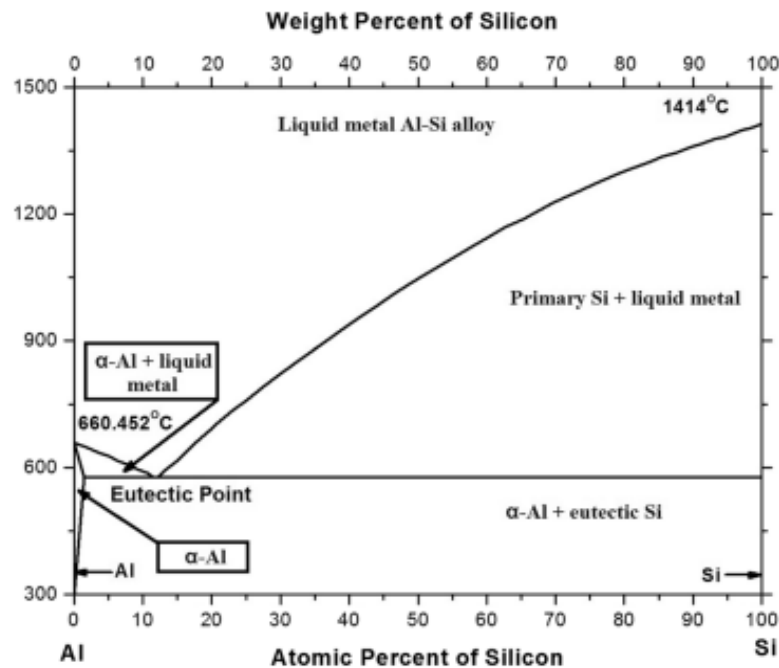


Figure 1.3 - Al-Si Phase Diagram

The phase diagram for Al-Si alloys is a binary diagram. The main point of this diagram is the eutectic point, that is where there is the solid-liquid transformation of the alloy. The eutectic temperature is the temperature at which the eutectic mixture is formed and it is 577°, and the eutectic composition is the composition of the alloy at this temperature, it is 12.5% of silicon.

During the solidification hypoeutectic Al-Si alloys, those with less than 12.5 % of silicon, are formed by α -primary phase dendrites with a lot of aluminium in the centre, surrounded by the eutectic Al-Si. When the temperature decreases the eutectic reaction takes place: a mixture of the α -solid solution of aluminium and silicon crystal is formed. In hypereutectic alloys, those with high silicon content, the crystals change shape and become more globular, due to the addition of other elements such as sodium. Thanks to this process the alloys improve their toughness and ductility. In alloys, silicon is usually used between 7 and 20%. Going beyond 20% would increase the temperature of the *liquidus* too much and lose the advantage given by this element, leading to greater brittleness of the material. As mentioned earlier, silicon gives a certain fluidity to the material. Fluidity is calculated as it follows: the alloy is melted at 800°C and cast in a spiral, if it travels a certain amount of spiral before solidifying then the alloy is fluid, otherwise it is

viscous. Fluidity is calculated in millimeters. Another key characteristic of silicon is that it decreases volumetric contraction. In fact, pure aluminium when it goes to solidify contracts by about 8%. By using instead an aluminium-silicon alloy with silicon at its eutectic point (at 12.5 %) the risk of shrinkage cavities decreases to 3.5 %. Silicon, therefore, tends to decrease the coefficient of thermal expansion and density, but if added in large amounts it tends to increase the brittleness of the alloy. Depending on the alloy where it is inserted, silicon has different effects. In summary, silicon provides these characteristics:

- Better castability and consequently better fluidity and less shrinkage from solidification
- Better mechanical strength
- Better wear resistance
- It determines the presence of a eutectic, low-T solidification point

1.3 Thermal properties

Aluminium is a very used material, not only because it is easy to find, but also because of its properties. Particularly this project is going to focus on his thermal properties. The thermal properties of materials are important because they influence how heat is transferred through them and how they respond to changes in temperature. Knowing them is very important for different reasons. In many applications, it is important to control the transfer of heat in order to maintain safe operating temperatures, prevent damage or failure of components, or optimize performance [10]. Understanding the thermal properties of components can help to minimize energy consumption and waste. They can also influence the choice of the material for particular applications. So studying them is very important for ensuring safety, reliability, and efficiency in various applications. Thermal properties refer to the properties studied on a material when it exchanges heat energy with another body or with the outside world. This heat transfer occurs because of the temperature difference between two bodies. In particular, it can be transferred in several ways:

- By conduction: when in steady state
- By convection: when one of the two bodies is fluid

- By radiation: when two surfaces not directly in contact, emitting the same heat, create a radiation network that allows the two bodies to exchange heat.

In particular four properties are going to be analyzed that are fundamental to this final project: thermal conductivity, thermal diffusion, heat capacity, and thermal effusivity. Before going to explain them in detail, there is one equation that ties them all together: the *Fourier law*. This law quantifies the heat transferred from one body to another. Consider, for example, a solid body with two flat faces that placed at a distance d , have two different temperatures T_1, T_2 , as in the figure 1.4.

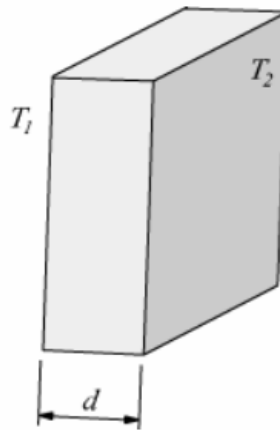


Figure 1.4 - Example of a solid

The temperature difference is the sufficient condition for heat transfer to occur. It might appear that there is a transfer of molecules or atoms, but since our example is solid, heat exchange will occur only through vibrational motions. This method of heat transfer can also be attributed to fluids, assuming it is not moving. The Fourier law succeeds in quantifying the energy moving from one body to another following this equation:

$$q = -A k \frac{T_2 - T_1}{d} \quad \text{Eq. 1.1}$$

Where q is the heat rate, A is the area where there is the transfer of the heat, k is the thermal conductivity (it is a constant that depends on the material), T_1 and T_2 are the temperature of the two surfaces ($T_1 < T_2$) and d is the distance between the two surfaces. The negative sign goes to indicate the direction of heat transfer that occurs

in the direction of the negative temperature gradient, that is, from the hottest to the coldest surface.

1.3.1 Thermal conductivity

Thermal conductivity is a property defined as a transport property in that it defines the amount of energy (heat) that is exchanged during a process across a surface A of thickness d [11]. From the Fourier equation cited above, it can be seen that thermal conductivity, defined by the letter k and denoted:

$$k = - \frac{d \cdot q}{A \cdot (T_2 - T_1)} \quad \text{Eq. 1.2}$$

Its unit of measurement is $\frac{W}{m K}$.

Its value is related to the physical composition of the material, so solid materials will have higher values than aeriform materials. Particularly when dealing with solid materials, electrical conductivity is formed by two factors, an electronic component and a lattice component. In fact, the particles that make up solids are very close to each other, energy propagates through two ways: the passage of free electrons k_e and lattice vibration waves k_l . Therefore, it can be said that:

$$k = k_e + k_l \quad \text{Eq. 1.3}$$

Moving from metallic to nonmetallic solid materials, the k_e component gradually decreases, while the lattice component increases. In quartz, for example, k is given almost entirely by the k_l component. In fluid materials, on the other hand, the intramolecular space is larger than in solids. As a result, the transport of energy between particles has less power. That is why thermal conductivity has smaller values. Following the rules of kinematic theory, however, it can be said that thermal diffusivity is directly proportional to:

- n , number of particles per unit volume
- \bar{c} , molecular velocity
- λ , the average distance a particle travels before colliding with another.

$$k \propto n \underline{c} \lambda$$

Although \underline{c} and λ are dependent on gas pressure, thermal diffusivity is not.

Thermal conductivity values usually increase proportionally to temperature and inversely to molecular weight. For aluminium and its alloys, values can vary from 70 to $230 \frac{W}{mK}$, depending on which alloying elements are present and in which quantity.

1.3.2 Thermal diffusivity

When it comes to thermal diffusivity it means the propagation of heat under non-stationary conditions, that is, conditions that change over time [12]. This property is intrinsic to the material in that it depends on characteristics exclusively of it. It depends on:

- density ρ ;
- specific heat c_p ;
- thermal capacity k .

It is calculated as:

$$\alpha = \frac{k}{\rho \cdot c_p} \quad \text{Eq. 1.4}$$

and it has unit of $\frac{m^2}{s}$.

In the formula the numerator is a value concerning the capacity to conduct thermal energy, while in the denominator the product $\rho \cdot c_p$ termed *volumetric heat capacity*, describes the ability to store heat. These are two opposite trends of the material. In every case the higher the thermal diffusivity is, the faster the heat propagates and the materials will respond quickly to changes in their environment. As with the thermal conductivity, also for the thermal diffusivity the metallic solids have higher values than non-metallic ones. For pure aluminium, the reference value is usually $97 \frac{mm^2}{s}$; when adding elements to create alloys, the thermal diffusivity usually drops, even to 44 as for Al-5Mg-2Si-Mn alloys.

1.3.3 Heat capacity

The heat capacity is a property typical of every material. It is an important physical property of a substance, as it determines how much energy is required to change its temperature. The definition is “*The heat capacity of a body or a system is the amount of heat required to raise the temperature of unit mass by 1°C*”. It can be classified into two types: specific heat capacity and molar heat capacity. The first one is the heat capacity per unit mass of a material and its unit is $\frac{J}{kg K}$. Diversely molar heat capacity is the heat per mole of a substance and it is expressed in $\frac{J}{mol K}$. The specific heat therefore depends on the mass of the body being studied and the temperature. The formula for calculating it is:

$$C_s = \frac{Q}{\Delta T \cdot m} \quad \text{Eq. 1.5}$$

Where C_s represents the heat capacity, Q is energy or the heat added, ΔT refers to the change in temperature and m is the mass. As mentioned before, heat capacity depends on various factors such as mass and therefore chemical composition and atomic structure. Solids generally have higher capacity than liquids or gases because they have a more packed structure and for this reason it requires more thermal energy to be raised by a given temperature change. Heat capacity depends also on temperature: when a temperature of the material is rising, its heat capacity decreases, because of the increasing thermal energy in its structure.

1.3.4 Thermal effusivity

The thermal effusivity is a typical thermal property of materials and is defined as the ability of any material to exchange heat with its surroundings [13]. It is calculated in this way:

$$e = \sqrt{\lambda \cdot c_p \cdot \rho} \quad \text{Eq. 1.6}$$

From the formula, therefore, it can be seen that its value depends on:

- λ , coefficient of thermal conductivity

- ρ , density
- c_p , specific heat capacity.

In the SI, the units of thermal effusivity is $\frac{W \sqrt{s}}{m^2 K}$.

It is a combination of the parameters explained earlier as thermal conductivity and specific heat capacity. The formula for effusivity takes these properties and provides a single parameter to describe the thermal diffusivity of a material. Effusivity in fact is very useful when comparing different materials using only one parameter. For example, a material with high thermal conductivity and low density has a higher effusivity than a material with low thermal conductivity and high density. Effusivity is a useful parameter for characterizing the thermal properties of materials. By taking into account multiple physical properties, it provides a simple way to compare and select materials for various thermal applications. So, the thermal properties of these alloys are influenced by several factors, including the composition of the alloy and the temperature at which the material is exposed. If the thermal properties of aluminum and silicon alloys are considered, it is clear that they have high thermal conductivity and this makes them a popular choice for different applications.

2. MICROSTRUCTURE

Before moving to the thermal properties of the sample, its structure should be defined. The sample studied at this stage is a sample cast together with the samples studied to define the thermal properties. In particular, the one studied has one side that has been in contact with the mould (marked with the red line in figure 2.1) and this can be seen from its not plain surface. It can therefore already be predicted that the microstructure of the sample will be different depending on which part is considered, close to the mould or more central.

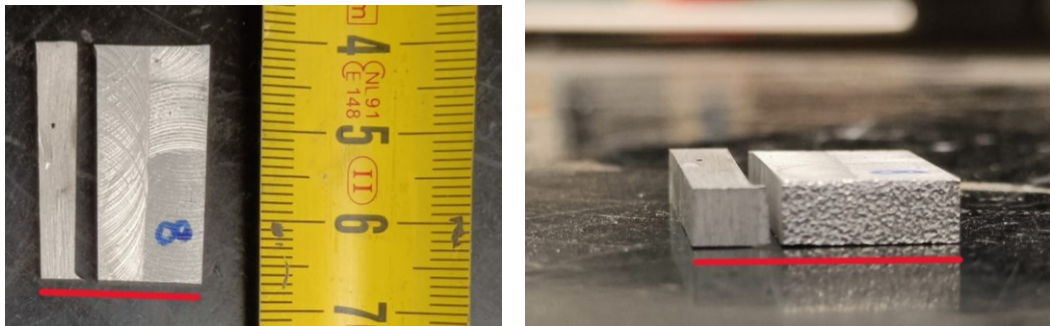


Figure 2.1 - Tested sample

2.1 Chemical composition

Firstly, the composition of the material is defined thanks to a spectrometer. This tool generates gaseous ions from the specimen. They form a reflected beam of light that can be transmitted in the visible region of the entire light spectrum. Depending on the recorded reflection angle, the spectrometer defines elements that are present in the sample's composition.

The average results obtained are shown in table 2.1.

Table 2.1 - Chemical composition of the sample

<i>Elements</i>	<i>Obtained values</i>	<i>Values of a EN 46000 alloys</i>
Al	84,82	Balance
Si	9,37	8,0 - 11,0
Fe	0,87	0,6 - 1,1
Cu	3,31	2,0 - 4,0
Mn	0,24	0,55
Mg	0,19	0,15 - 0,55
Zn	1,03	1,2
Cr	0,03	0,15
Ni	0,05	0,55
Ti	0,04	0,20
Pb	0,08	0,35
Sn	0,01	0,25

The column on the right shows the values of the different elements according to the standards of an EN 46000 aluminium alloy [14]. Looking at the data, it can be stated that the alloy studied belongs to this category. In the Italian codification, however, these types of alloys are defined as UNI 5075.

3.2 Microstructure

Before studying the microstructure of the material, the analysed specimen has to be created. There are four basic steps to follow: cutting, embedding, honing and lapping. Initially, a longitudinal portion of the specimen is cut with the cutting machine. To make the specimen more stable, the just cut piece is 'encased' in a cylinder of black phenolic resin. With this process, only the face of interest for analysis is left uncovered. Next, to make the surface with a low roughness so that the microstructure of the material can be seen under the microscope, the sample is sanded. The sanding is done using abrasive papers with different grits (e.g. 180, 600, 1200 and 2400 grit). The last step is lapping, which is used to clean the sample. It is very similar to polishing in terms of both process and machinery, but instead

of using abrasive paper, diamond paper is used. Once these steps have been carried out, the sample is obtained (figure 2.2) and it can be analysed.

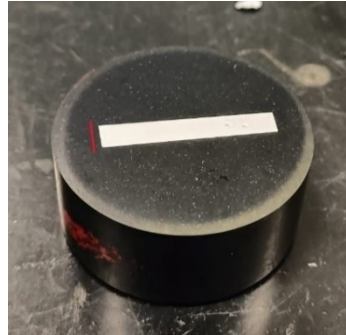
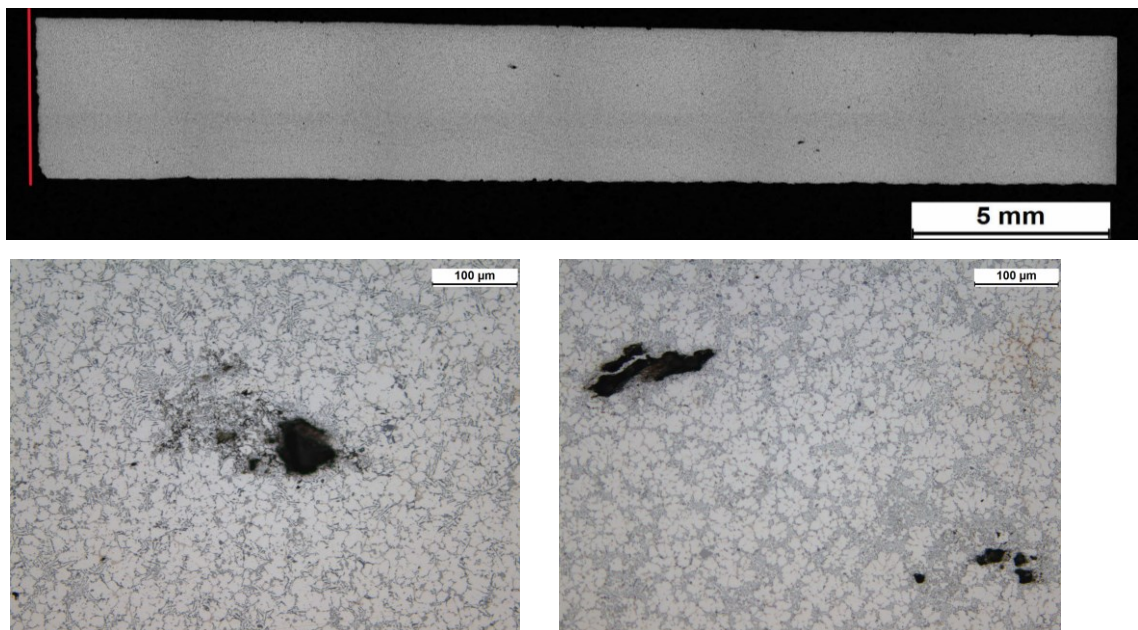


Figure 2.2 - Sample ready for the analysis (the red part is the one near to the mould)

To study the microstructure two methods are used: one with the light microscope and the other with the scanning electron microscope (SEM). The first one generates the images thanks to light reflecting off lenses, while the SEM generates the image thanks to an electron beam that is thrown onto the material in a vacuum chamber; the interaction between the electrons in the beam and the atoms in the material prompts them to emit signals, giving information on both topographical (SE) and chemical (BSE) analysis. This final project will focus only on chemical analysis. The first analysis is done with the light microscope. The amount of defects is defined initially using the highest magnification. It can be seen in figure 2.3 that there aren't so many structural defects.



*Figure 2.3 - Up: full cross-section (red line is the side next to the mould),
Down: zoom on oxide defects on the surface*

At this point, magnifications are increased by dividing the specimen into two zones, the one near the mould (figure 2.4) and a more central zone (figure 2.5).

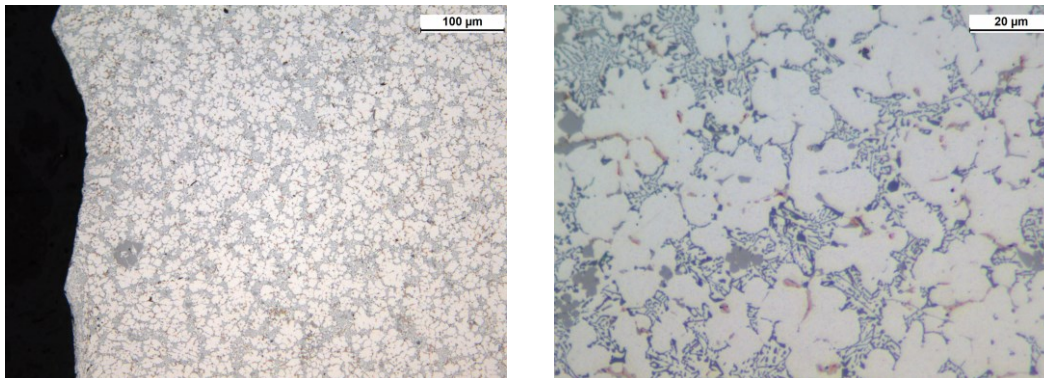


Figure 2.4 - Frame next to the mould, on the left magnitude 100x, on the right magnitude 200x

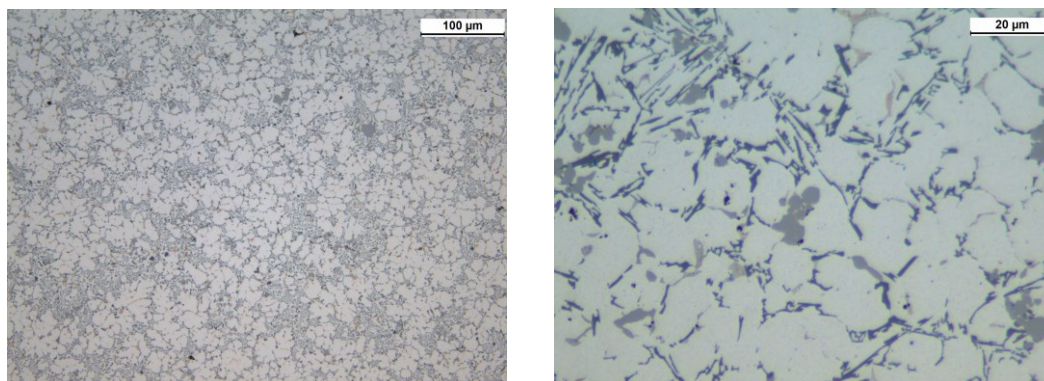


Figure 2.5 - Frame in the central part of the sample, on the left magnitude 100x, on the right magnitude 200x

From these images, it can be seen that the eutectic material forms a lamellar structure, these lamellae made of silicon are more elongated in the central area than in the area near the mould, where they are shorter. Near the mould the material cools faster and the eutectic tends to break apart, compared to the slower cooling in the central part. Another difference that can be seen is that the primary cells of the aluminium are larger in the center than near the mould.

The figure 2.6 shows the material constituents.

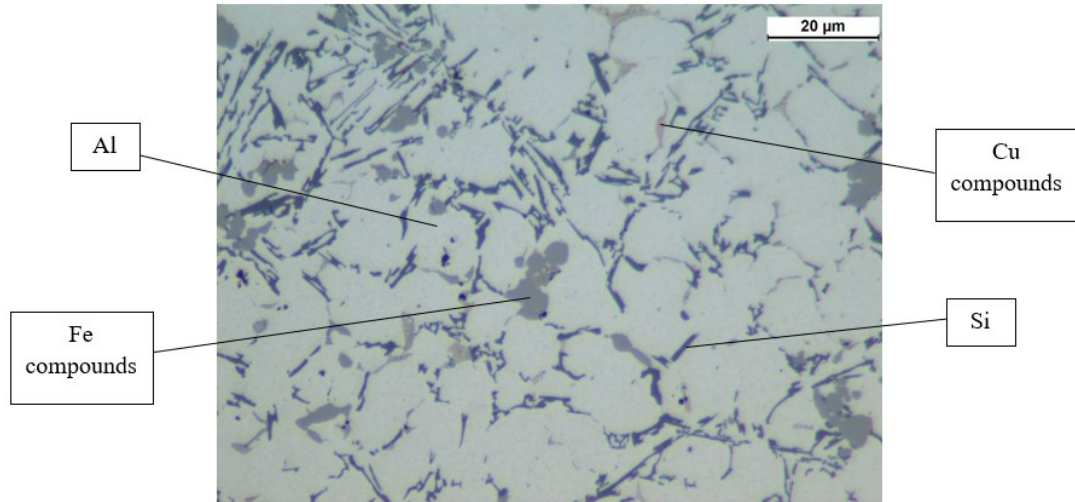


Figure 2.6- Material constituents

In the figure, the white part that covers the majority of the frame is aluminium, while the dark grey lamellae are silicon. Other compounds such as copper and iron are also visible.

In order to identify the elemental composition, the SEM is used. This microscope not only has a higher magnification than the optical microscope, but also allows the percentage of elements present in a specific area of interest to be obtained via a tool. The images obtained are in grey scale, the colour difference is based on their atomic number (Z): materials with a lower Z are darker and those with a higher Z have a colour near the white (figure 2.7).

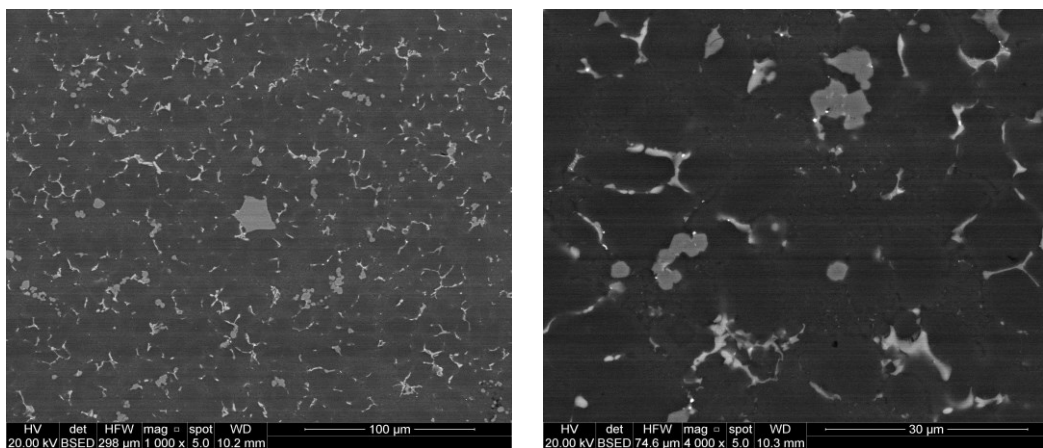


Figure 2.7 - SEM: on the left magnitude 1000x, on the right magnitude 4000x

From the figure 2.7 the matrix seems to be all the same material, the colour in fact is more or less homogeneous over the whole frame. Otherwise the figure 2.6 shows that in the matrix there are Al and Si. From the image taken with the SEM it is hard to distinguish the two elements very well as they have a very similar atomic number, $Z_{Al} = 13$ and $Z_{Si} = 14$, and thus a similar colour. In the figure there are compounds lighter than the matrix and some white points. To analyse these compounds energy-dispersive X-ray spectroscopy, EDS, is used. It is a tool implemented in the microscope used to analyse the chemistry of the sample.

Different types of compounds are found. To simplify the description it can be said that there are three different categories:

1. Fe- α , that is of general chemical formula $Al_{15}Si_2(Fe, Mn, Cr, Cu)_3$. These types of compounds are usually grey and they have a polygonal form. They are particularly characterised by the presence of Cr as can be seen in the figure 2.8.

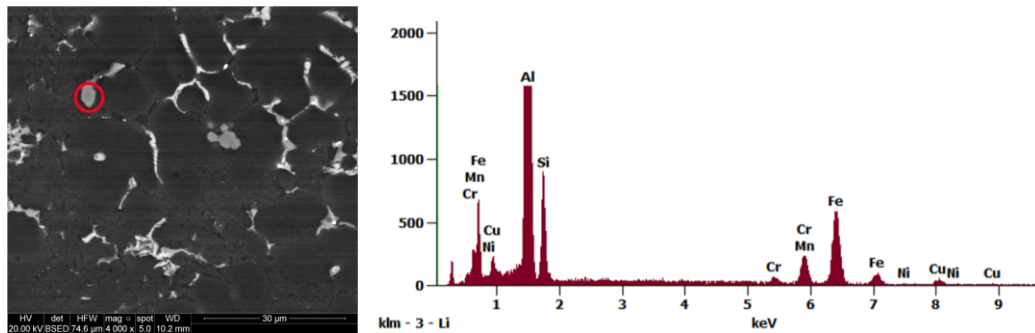


Figure 2.8 - SEMI: on the left magnitude 4000x, on the right the spectrum of the components of the circle

The table 2.2 shows the weight percentage of the elements found in the particle. The spectrum and the chemical composition are referred to the circled compound in the figure 2.8.

Table 2.2 - Chemical composition of the first compound found

Elements	Al	Si	Cr	Mn	Fe	Ni	Cu
Weight (%)	63,97	8,37	1,21	6,20	17,73	0,47	2,05

2. AlCu₂, these types of compounds are lighter than the first one. They can have different shapes: more rounded like the one marked by the red circle on the left in figure 2.9 or more elongated and fragmented like the one highlighted in the green circle on the right.

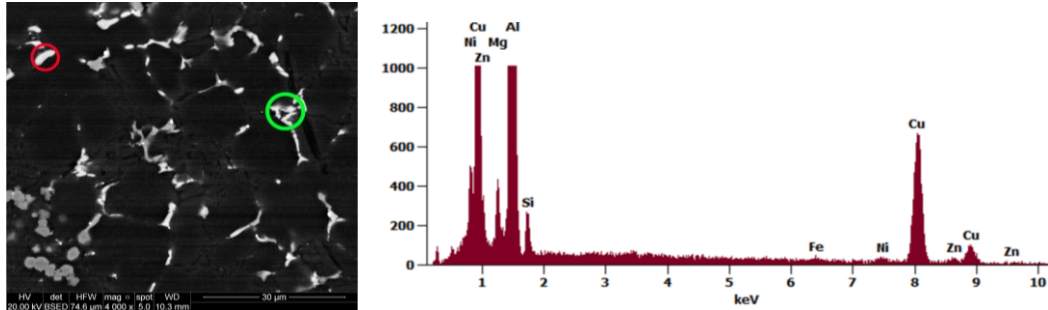


Figure 2.9 - SEM2: on the left magnitude 4000x, on the right the spectrum of the components of the red circle

The table 2.3 shows the weight percentage of the elements found in the particle in the red circle.

Table 2.3 - Chemical composition of the second compound found

Elements	Al	Mg	Si	Cu	Zn	Fe	Ni
Weight (%)	73,63	0,92	1,61	19,59	0,44	0,29	0,52

3. An intermediate phase between the previous two, compounds containing both Cu and Fe are present. From the quantities shown in the table 2.4, it is hypothesised that they could be Al₁₅ Si₂ (Fe, Cu)₃ compounds [15]. Their composition is very similar to the composition of Fe- α , but with more copper.

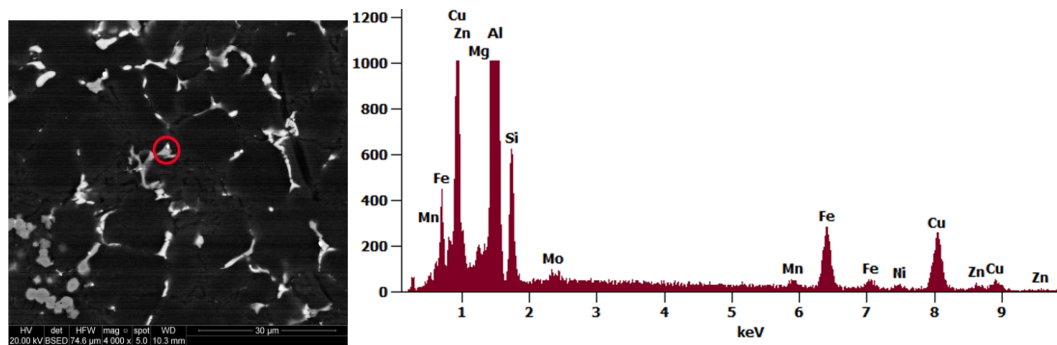


Figure 2.10 - SEM3: on the left magnitude 4000x, on the right the spectrum of the components of the red circle

Both the spectrum in the figure 2.10 and the chemical composition in the table 2.4 are referred only to the grey part in the red circle.

Table 2.4 - Chemical composition of the third compound found

Elements	Al	Mg	Si	Mn	Fe	Ni	Cu	Zn	Mo
Weight (%)	71,65	0,48	5,24	0,99	6,91	0,85	12,21	1,24	0,44

The white dots that can be seen in all different figures are now analysed.

As the figure 2.11 shows, it is found out that they are lead. They are much lighter in colour than the others because Pb has an atomic number equal to $Z_{Pb}=82$, so it is heavier than the other elements.

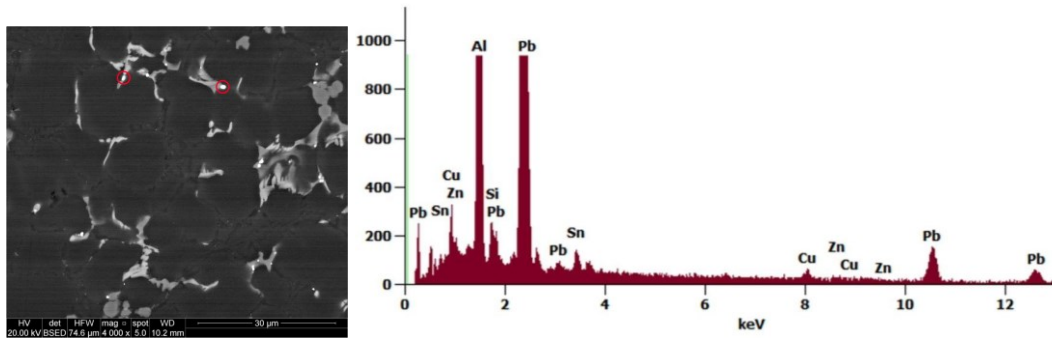


Figure 2.11 - White points: on the left magnitude 4000x, on the right the spectrum of the components

Analysis with the spectrometer revealed a large amount of zinc, this was one of the reasons why it is decided to do the chemical analysis with the SEM. Studies carried out show that the zinc is present in the matrix, in the compounds with copper and in the compounds described in the point three of the previous list, thus the compound made by iron and copper. It is not present in alpha iron.

After this analysis, considering that the alloy studied has a high amount of iron and copper compounds, it is decided to quantify the average area occupied by these two compounds by taking an area of 1 mm².

Firstly, copper compounds percentage is quantified: the sample is submerged in an aqueous solution of 25% HNO₃. The sample was immersed for about 40s at 70 °C. In this way, the copper compounds are burnt and they become darker when viewed

under the microscope, as in figure 2.12. The average area occupied by copper compounds is: $2,94 \pm 0,34\%$.

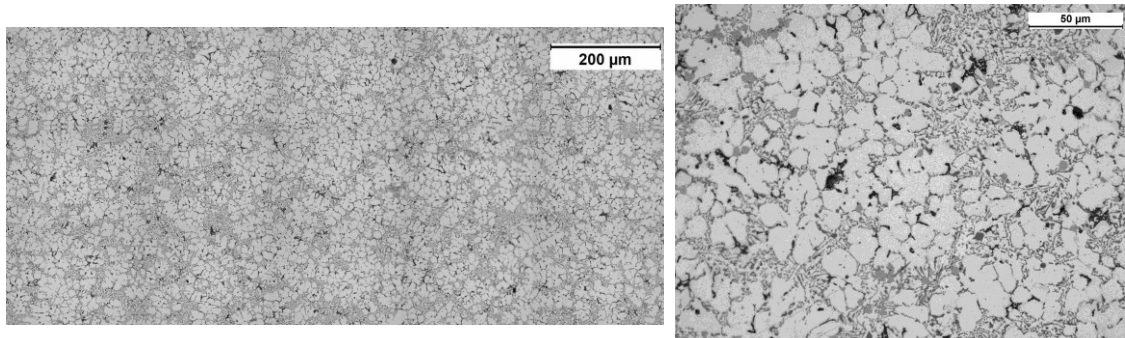


Figure 2.12 - Sample after chemical attack (Cu), magnitude 50x (left) and magnitude 250x (right)

After lapping and cleaning the sample to remove the layer attacked by the previous reaction, the iron compounds percentage is quantified. The sample is always submerged in an aqueous solution of 20% H_2SO_4 . The sample is immersed in the solution for about 50s at 70°C . In the figure 2.13 the Fe compound can be seen, darker than the other elements.

The average area occupied by iron compounds is: $2,36 \pm 0,44\%$.

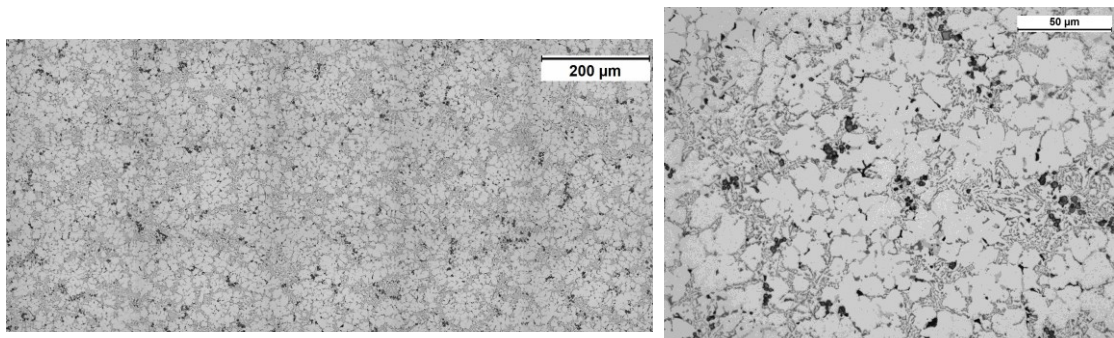


Figure 2.13 - Sample after chemical attack (Fe), magnitude 50x (left), magnitude 250x (right)

After quantifying both copper and iron compounds percentage, the sample is cleaned again.

It is decided to visualise and quantify the amount of eutectic, as before, in terms of average area considering a frame of 1 mm^2 . The Keller reaction is used to highlight these parts. The sample is immersed in a solution composed of 95% H_2O , 2,5% HNO_3 , 1,5% HCl and 1% HF . Figures 2.14 are obtained with the microscope.

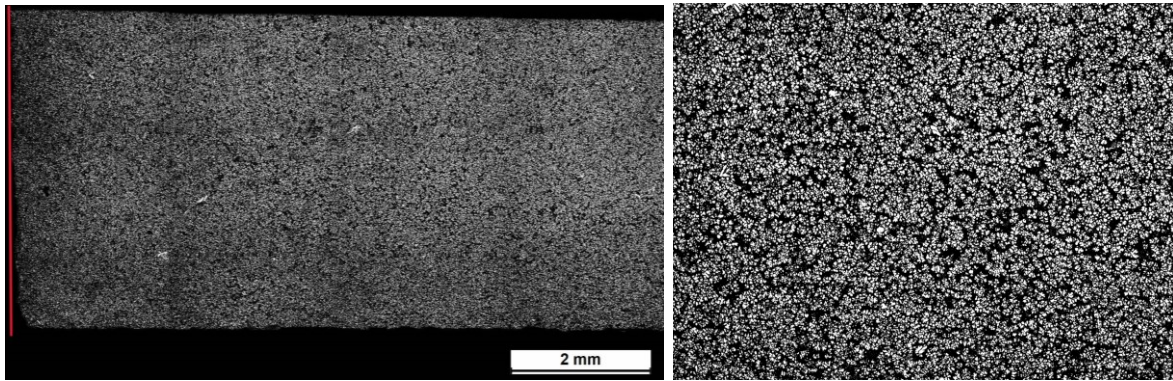


Figure 2.14 - Sample after chemical attack (eutectic) (left), magnitude 250x (right)

From the overview (Figures 2.10) it can be noticed a thickening of eutectic (black part) in the left part, that is the part in contact with the mould. A smaller percentage, but still with an even distribution, in the central part can be seen.

The area occupied by the eutectic is more or less the half of the sample, it is equal to: $52,21 \pm 1,93\%$.

3.3 Hardness test

As a final test, the hardness test was carried out. The hardness of a material is defined as its resistance to penetration by a harder material. The hardness test is a simple, quick and non-destructive test. It is usually done for production and product quality control. Although there are different methods to define hardness, the test that is performed is always the same. A body of higher hardness is used and the load is applied through this body. It is applied statically and perpendicular to the surface of the sample to be analysed. The difference between the different hardnesses also depends on the type of indenter. For this sample, the Brinell hardness test was used, which uses a hardened steel ball with a diameter of 2.5 mm as indenter. The load used is 62.5 kg. It is applied for about 30 s, giving the alloy time to deform.

The values obtained are:

Table 2.5 – Hardness test

	Diameter [mm]	HB_{2,5/62,5}
Test 1	9,2	91
Test 2	9,3	89
Average		90

Hardness tests were carried out in the central part of the specimen as shown in figure 2.15.



Figure 2.15 - Hardness test

3. TOOLS AND METHODS

3.1 Hot Disk

The instrument used for measurements is a tool from Hot Disk technology [16, 17, 18, 19, 20, 21, 22]. Hot Disk is a technology that allows several rapid, nondestructive tests to be conducted to calculate the following properties on different materials:

- Thermal conductivity
- Thermal diffusion
- Heat capacity
- Thermal effusivity

This technology is very flexible and does not require special geometries to test samples. Several materials can be tested with this instrument, not only metal materials, but for example also the properties of wood or milk can be calculated. These instruments originated from a Swedish family-owned company in Gothenburg, where the headquarters are still located in the 1980s. Hot Disk takes place in a network that allows it to have a global market, but also to expand into application fields as diverse as aerospace, civil engineering... Different models of testing machines have been introduced in the market since the 2000s, but the one that has been used in our case is the Hot Disk model TPS2500 S (figure 3.1).

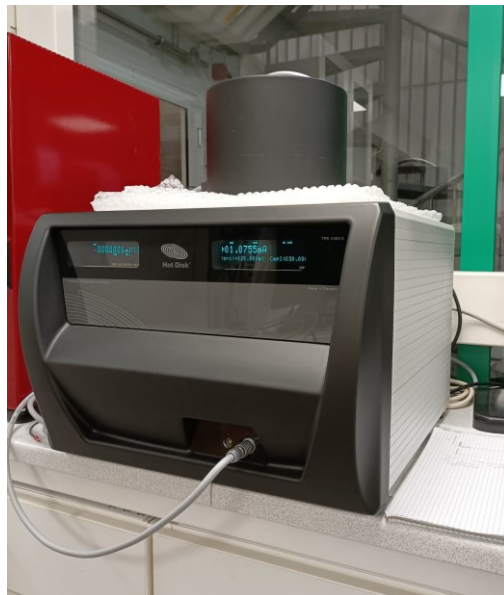


Figure 3.1 - TPS2500 S

This instrument was introduced in the market in 2007, it is the most widely used in the market nowadays, as it can take several measurements at the same time, one measurement takes a minimum of 1 second, and can be used with different materials both solid and liquid. The tested materials can be isotropic, and thus have all properties the same regardless of direction, but also anisotropic. Materials with surfaces on which to do the tests of different sizes such as sticks or plates can be used. In the table 3.1, the specifications of the model that was used for this project are reported:

Table 3.1 - Characteristic of TPS2500 S [23]

Thermal conductivity	0,005 to 1800 W/mK.
Thermal diffusivity	0,01 to 1200 mm ² /sec.
Thermal effusivity	20 to 55000 W ^{1/2} /m ² K.
Specific heat capacity	Up to 5 MJ/m ³ K.
Measurement Time	1 to 2560 s
Reproducibility	Typically better than 1%.
Accuracy	Better than 5%.
Temperature range	-253 °C (20 K) to 1000 °C.
Main instrument	Environmental
With oven	Up to 750 °C (1000 °C oxygen free).
With circulator	-35 °C to 200 °C.
Power requirements	Adapted to the mains voltage of the country of use
Smallest sample size	0,5 mm × 2 mm diameter or square for mass testing.
	0,042 mm × 8 mm diameter or square for slab testing.
	5 mm × 2.5 mm diameter or square for one-dimensional testing.
	0,01 mm × 22 mm diameter or square for thin-film testing

Since 2008 this model follows all ISO 22007-2 standards, where ISO (International Organization for Standardization) is a federation of national standard bodies for all the world [24, 25]. The model is suitable for the standards that have been defined for the determination of thermal conductivity and thermal diffusivity. In particular,

this standard has been defined as applicable to plastics, but it can also be defined for many other materials of different forms. To allow testing samples of different geometries, materials and properties, there are some modules that measure different properties in different ways:

- *Isotropic*: This module is used to calculate the thermal properties of different samples, that can be solid, powder, liquid... The values obtained are an average value of the sample volume.
- *Anisotropic*: It is used for materials that have properties that change with direction. Usually properties are calculated in 2D, for 3D an additional sensor is needed. As with isotropic materials, measurements can be performed on either one side or two, but for greater accuracy it is recommended to perform tests on both sides.
- *Slab*: Metals, semiconductors and graphite are the samples of choice for this module. As input data there are the sample thickness, measurement time and output power. It is always convenient to use two halves of the sample for measurements.
- *Thin film*: Used in the case of thin insulators, in the range from 20 to 500 μm . As for the slab method, two halves are required.
- *Low Density/High Insulation*: This method is used for very low conduction materials, so the heat loss through the sensor medium becomes significant. In this way the heat loss can be corrected.
- *One-dimensional*: High conductivity should be one of the main characteristics of the materials used in this module, and for this reason the selected samples are limited. In this case the samples are very little rod-shaped samples. The diameter of the sensor must be as small as the diameter of the sample.
- *Structural probe*: It is used to test the average heat capacity as a function of probing depth. This tool is usually used for quality control testing.
- *Single-sided test*: With this method, properties are calculated using the sensor between two samples but also the test is done on one side only. As a result of testing on one side there will be less accuracy. For this reason with

this method it's very important to use an insulating support, to fight the asymmetric heat flow that there is around the sensor.

- *Specific heat capacity:* This test is done on any type of material, being it solid with variable surfaces, liquid, etc. It is a sensitive measurement because the material is placed in an insulated gold container and heated flat, allowing a more accurate calculation of specific heat. Measurements are made on individual samples.
- *Automation:* With this feature, all measurements can be controlled remotely.

These are all add-ons; the only standard measurement is isotropic. For this project different methods have been used to find the best one, based on the needs of the analysis. Briefly, the measurements are taken by means with a sensor that is placed between two pieces of the material to be tested. The temperature in the sensor gradually increases and in this way the thermal properties can be calculated. The choice of the sensor is very important, in fact there are many types of sensors; it can be round or rectangular, based on the form of material that needs to be studied. In our case the sensor would be round as in the figure 3.2.



Figure 3.2 - Sensor Kapton C5501

The round sensor is usually created with two spirals of nickel because of its electrical conductivity and its high coefficient of resistivity. The spirals are located between different types of materials, the choice of these materials depends on the temperature at which measurements should be made. Kapton is used for processing

at low temperatures up to 500K, while Mica is used for higher temperatures up to 1000K. These films are very thin, they are on the nanometer scale. Depending on the material, the sensor is able to sustain different temperatures, but also the sensor's lifetime will change. In fact when Kapton is treated in a good way, it can be used for more experiments, while Mica has a shorter life as higher temperatures are usually used. To make measurement easier and with high accuracy, the samples must not be thinner than the radius of the sensor. In this experiment, at first the isotropic module was used, that is the simplest process, requiring only a few input data. If desired, one can also test a sample whose material properties are unknown. Both one-sided and two-sided measurements can be carried out. The following points are the list of parameters required for the first measurement:

- *Sample identity*: The sample material, for example metal, aluminium, polymer, ceramic, specifying if the composition needs to be tested.
- *Available Probing Depth*: It defines how deep into the sample the generated heat is calculated. This parameter is connected to the thermal wave. Due to the fact that the sensor is considered immersed in an infinite plane, the thermal wave will never reach the boundaries of the sample and in this way it will not be affected by external agents. During the measurements we therefore take into account the probing depth which is calculated in the following equation:

$$\Delta p = 2 \cdot \sqrt{k \cdot t} \quad \text{Eq. 3.1}$$

Where k is the thermal diffusivity of the material; t is the measuring time of the experiment and 2 is a constant. From this formula the larger the samples, the easier taking measurements will be. So, the distance between the sensor and the nearest outside boundary should always be larger than probing depth.

- *Disk Type*: As written before there are many types of sensor (disk type) and it must be chosen according to the measurements.
- *Radius of Disk*: The specimens studied are homogenous metals, so the sensor has the radii between 3.2 and 15 mm. The sensor has to be smaller than the specimens, in this way the boundaries are larger and the possibility

of external influences are reduced. The diameter of the specimens has to be more than twice the diameter of the sensor. Choosing the sensor is good, do not forget that there is a minimum time for the experiment. For measuring the thermal diffusivity and conductivity the time should not be less than 5 seconds, in this way data will be more precise.

- *TCR (Temperature Coefficient of Resistivity)*: It is a coefficient, thanks to which the variation of the temperature can be calculated, recording the resistance as a function of time, and then calculating the thermal transport properties. The positive aspect is that nickel has a TCR over a large range, but if the transition is in the range from 350 °C to 400°C, the experiment must be done very carefully.
- *Initial temperature*: It is the temperature of the room.
- *Output Power*: The electrical power that is used to heat the sensor during the experiment.
- *Measuring Time*: It is the measurement time, it describes how long it takes the machine to obtain the data.

In the figure 3.3 it is shown what the screen for entering parameters looks like.

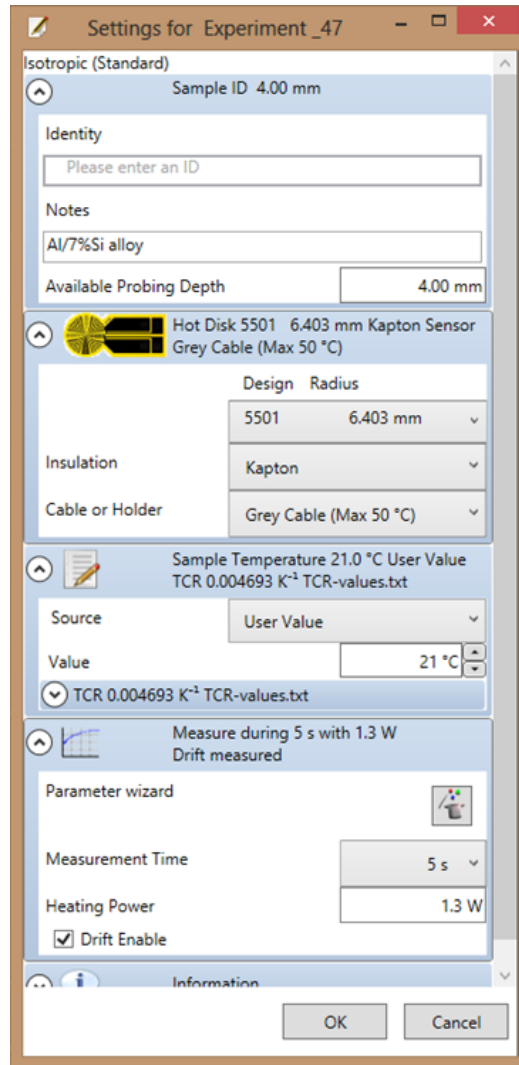


Figure 3.3 - Isotropic methods parameters

The last two parameters are very important. They can be chosen by a list that is given by the software called “*Parameter Wizard*” if it is the first experiment and the thermal properties of the material are not well known; or if similar tests have been done before with this material, those parameters can be used to get precise and reliable measurements. Another time that is fundamental for this measurement is the *characteristic time*; it is the total time of the transient recording. It is described by this equation:

$$t = \frac{a^2}{k} \tag{Eq. 3.2}$$

where a is the radius of the sensor and k is the thermal diffusivity of the material. This time is directly proportional to the size of the sensor, so the bigger the sensor,

the longer the experiment will last. The ratio between the total time of the experiment and this time described above should be from 0.5 to 1. In order to assure that measurements are taken correctly 15 minutes should usually elapse between experiments, so the temperature can return as it was in the beginning and the experiment can be repeated.

Another method to be described is the *SLAB method*. This is very similar to the standard method that was used for the first measurements but with two main differences. First of all, there has to be a material with low thermal conductivity to protect the lateral surfaces of the slab. In this way heat losses might be reduced. Then knowing that the heat propagates in the slab's direction, which is assumed as an infinite plane. It is used for situations with the following ratio between the slab thickness and the sensor radius:

$$0.03125 < \frac{h}{a} < 0.79836$$

For the second measurement, the same parameters were used as for the first, with only two additional features:

- *Available Radial Probing Depth*: It is the same probing depth that was explained before, but in this case it refers to radial directions.
- *Thickness of Slabs*: How thick the slabs are.

Thanks to the slabs, heat doesn't dissipate, so when in order to repeat the measurement there will be more waiting time between measurements to reach the initial temperature in the samples as opposed to the previous method.

As seen briefly before the *Thin Film Method* is used for studying the thermal conductivity of thin samples. The experiment is carried out like the other methods, with the same inputs, only with the addition of defining the thickness of the film. The temperature drop during measurements becomes constant. This is due to the power, thickness and area of the insulating material, all of which are constant. It is essential to define these parameters well in order to obtain satisfactory results. With this method, the thermal conductivity can be obtained using the following formula:

$$\frac{P}{2} = A \cdot \lambda \cdot \frac{\Delta T}{\Delta x} \quad \text{Eq. 3.3}$$

Where P is the output of power, A is the area of the samples, λ is the thermal conductivity of the thin samples, ΔT is the temperature difference and Δx is the thickness of the coating.

With this method, the thermal conductivity of the thin film is defined, but at the same time the thermal properties, such as thermal conductivity, thermal diffusivity and specific heat, of the background (that is an isolation material for the film) are calculated. This method is very sensitive, as it calculates an apparent thermal conductivity, so even the smallest air gap or how the thin sheets of coating are made can influence the results.

3.2 Transient Plane Source

The Transient Plane Source (TPS) is the one method that is used in the Hot Disk [26, 27]. It is a new method that is used to determine the thermal diffusivity and the thermal conductivity of materials. This technique is used in a variety of fields, such as materials science research, quality control in manufacturing, and characterization of insulation materials. The TPS works by measuring the temperature rise of a sample and the resulting heat flow into the surrounding material. The temperature is monitored over time as heat is applied to the sample thanks to a sensor that is applied in the middle of two pieces of the sample. The thermal conductivity of the sample can then be calculated based on the temperature rise and heat flow. The TPS method is based on the principle of heat transfer, and takes into account the heat flow into the sample, the heat flow into the surrounding material, and the temperature difference between the heater and the temperature sensor. One of the benefits of the TPS method is its high accuracy and precision. In fact, the sample and the temperature sensor ensure that the heat flow into the sample is accurately measured. This leads to highly accurate and precise thermal conductivity measurements. Another benefit of the TPS method is its versatility. It can be used to measure a wide range of materials, from metals and ceramics to polymers and composites. This versatility makes it a valuable tool for materials science research, as it allows researchers to study the thermal conductivity of a variety of materials. So the Transient Plane Source method is a highly accurate and precise thermal

measurement technique that is widely used in a variety of fields. Its versatility and accuracy make it an important tool for materials science research, quality control in manufacturing, and characterization of insulation materials. After this general overview of this method, more specific aspects of how the measurements are made can be described now.

3.3 How to measure

Before starting the measurement, the correct sensor must be chosen, considering the material used and the temperature at which measurements will be taken. After this, the materials and the sensor can be placed in the support as in the figure 3.4 [28].

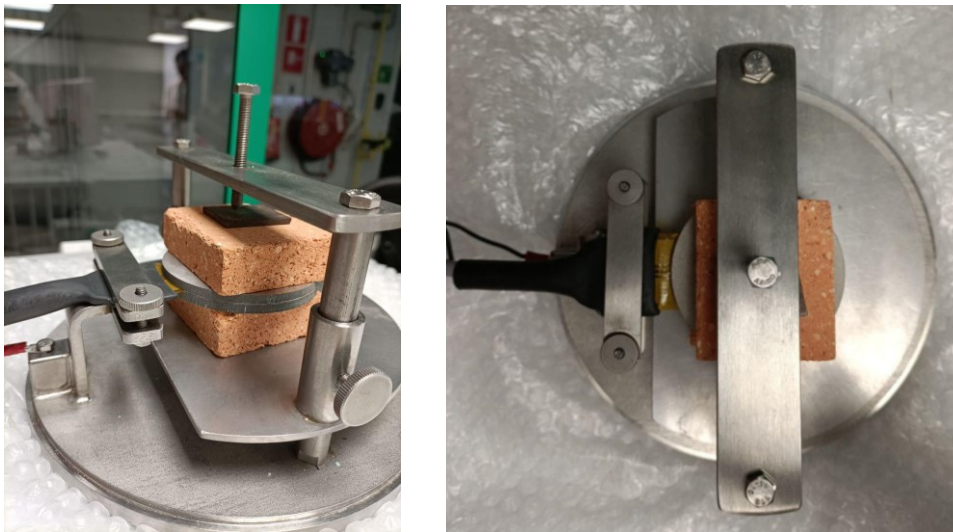


Figure 3.4 - How to position sensor and specimens

Firstly the sensor is placed on his special support, in a way that the double spiral is located in the center of the machine, under the steel bridge. After that, the samples are positioned; one under the sensor and one above it. Then, the piece that is under the sensor is raised in the way that the top plane surface is located at the same level of the sensor. In this way the sensor will be between the specimens. Usually, a piece of metal is used on top of the pieces, so that the clamping screw in the steel bridge can hold the pieces in place without damaging them. It can also ensure that the clamping pressure is unidirectional. It is mandatory that pieces are clamped firmly together to avoid the presence of an air gap into the system. If there is an air gap, it could cause overheating and damage the sensor. After this everything is closed

under a cylindrical cover. The location where the measurements are done has to be a stable environment, isothermal, no vibrations and have constant humidity. The samples are ready for measurements. After positioning the sensor and the specimens the parameters explained before have to be written in the software. The experiment can now begin. At this point the temperature starts increasing thanks to the current that is passing through the spirals. When there is electricity in those spirals, there is also a resistance, and it can be calculated in the following equation:

$$R(t) = R_0 \{1 + \alpha [\Delta T_i + \Delta T_{ave}(\tau)]\} \quad \text{Eq. 3.4}$$

Where R_0 is the resistance of the sensor at $t = 0$, α is TCR, ΔT_i is the difference of temperature in the insulating surfaces that cover the sensor and $\Delta T_{ave}(\tau)$ is the difference of the temperature on the other side of the insulating surface.

ΔT_i can be considered as a constant, because the time it takes to be equal to 1 because it is defined as:

$$\Delta t_i = \frac{\delta^2}{k_i} \quad \text{Eq. 3.5}$$

Where δ is the thickness of the insulating layers and k is the thermal diffusivity of the material.

$\Delta T_{ave}(\tau)$ can be estimated with equation 3.6:

$$\Delta T_{ave}(\tau) = \frac{P_0}{\pi^2 \cdot a \cdot \Lambda} \cdot D(\tau) \quad \text{Eq. 3.6}$$

Where P_0 is the power of the sensor, a is the total radius, Λ is the thermal conductivity of the material and $D(\tau)$ is a time that depends on

$$\tau = \sqrt{\frac{t}{\theta}} \quad \text{Eq. 3.7}$$

in which θ is the characteristic time.

At the same time to the passing of currents, the machine registered the resistance increase as a function of time. As written before the sensor must be placed between

two surfaces larger than its size, in order to be able to do the measurements correctly. The sensor must be in an infinite medium, that way as soon as there is an external influence of other materials, measurements will no longer be taken. During the experiment the software records 200 data points. When the experiment finishes the software returns two charts, called “*Drift*” and “*Transient*”.

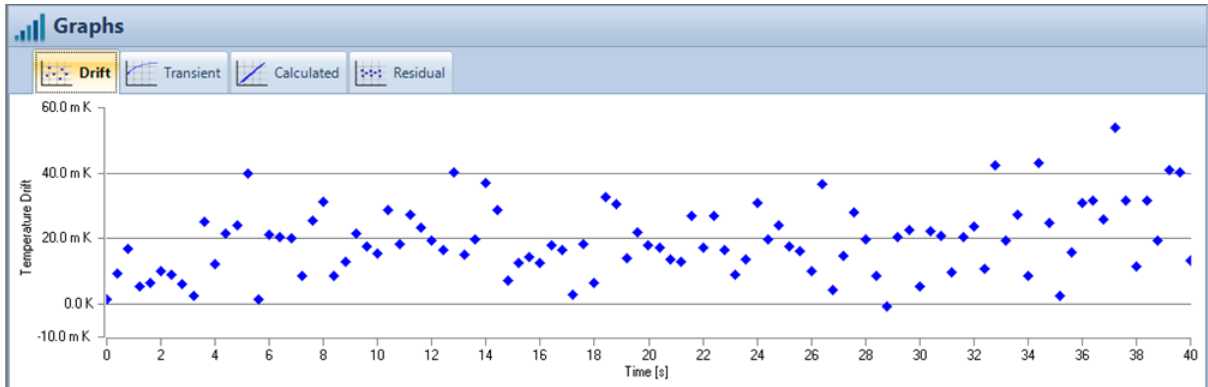


Figure 3.5 - Drift Chart

The *Drift* is a chart that shows the Temperature drift against Time (figure 3.5). It shows how the temperature of the sensor varies before heating. Variations must be minimal, if they are present it means that the samples are still cooling down.

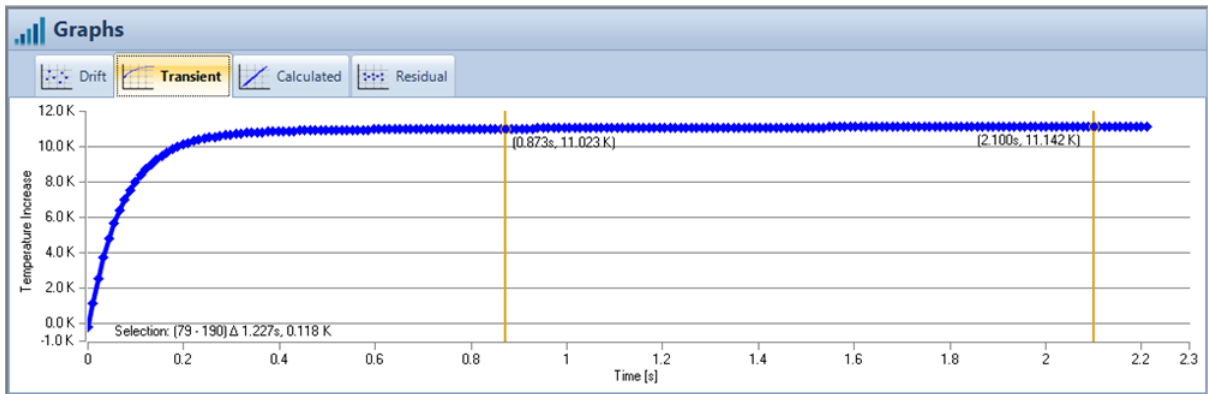


Figure 3.6 - Transient Chart

The *Transient* is a graph that shows the sensor temperature increases with the passage of time (figure 3.6). The line has to increase in an even way. If it is not like this, it is better to try again to take the data. Thanks to two cursors specific data can be selected for calculating thermal properties. The data at the beginning of the recording might be influenced by the heat capacity of the sensor and the data at the

end are affected by the limited size of the sensor. For these reasons data are not usually taken at the beginning and at the end. It usually brings at least 20 points to get reasonable statistics. After selecting the data, the thermal properties can be calculated. During the experiment the temperature rises directly with the power delivered to the specimens from the sensor. To compensate for the power variations caused by the heat capacity of the sensor the *Fine-Tuned Analysis* is used. In this procedure parameters are calibrated very precisely in order to align with specific observations. At the end, the calculations lead to the following charts (Figures 3.7 and 3.8).

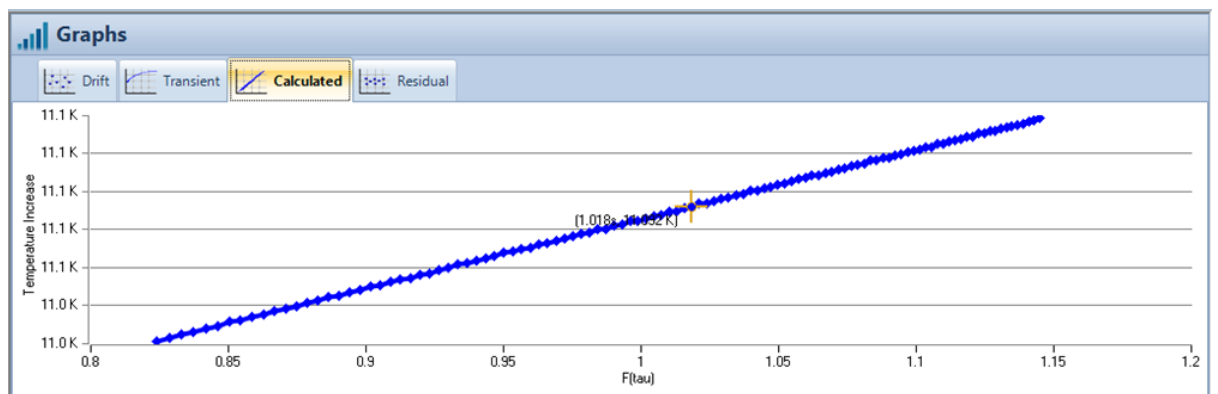


Figure 3.7 - Calculated Chart

The first one is with the Temperature Increase on the y-axis and $F(\tau)$ on the x-axis (figure 3.7). The temperature in fact can be expressed as a linear function of $F(\tau)$ that is a dimensionless time function. A straight line should appear in this graph, showing that the two dimensions are directly proportional. From the slope of the line the thermal conductivity can be calculated.

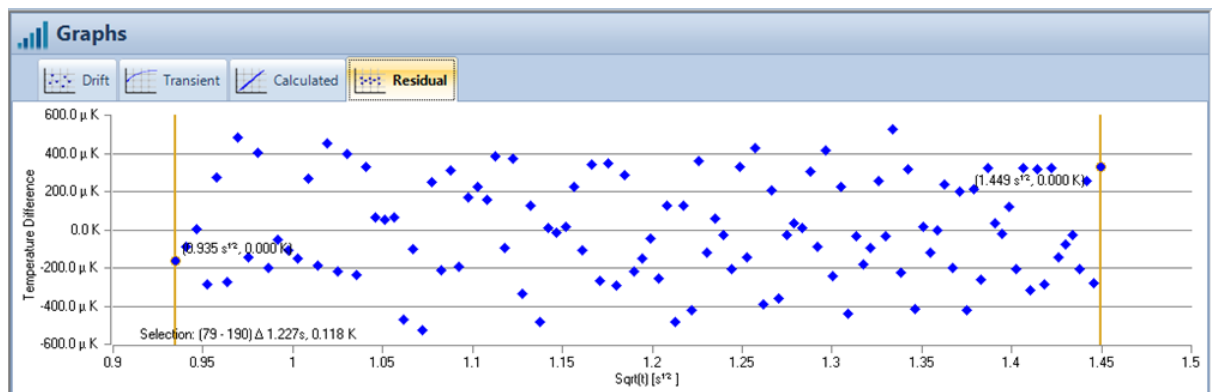


Figure 3.8 - Residual Chart

The other chart is a scatter plot with the Difference Temperature versus Square root of time (figure 3.8). It has to be random, this indicates that the calculations aren't disturbed by any lateral sample surfaces. If it is not like this then a new set of data should be chosen. If the chosen data set excludes more than 100 final data points, it is better to repeat the measurement by decreasing the *Measuring Time*. The selected data may be incorrect and it can be seen once the thermal properties have been calculated, even if the graphs seem correct. All the properties are described in a table.

In this table the reported numeric results are related to:

- Thermal conductivity
- Thermal diffusivity
- Specific heat
- Thermal effusivity
- Probing depth
- Temperature increase
- Temperature drift
- Total temperature increase
- Total/Characteristic time
- Time correction
- Mean deviation
- Disk resistance

The checkpoint that must be controlled are probing depth, temperature increase, total temperature increase, total/characteristic time and mean deviation. The probing depth has to be less than the available probing depth that was written at the beginning of the measurements. The ratio between total and the characteristic time has to be between 0.33 and 1. The mean deviation represents the noise of the measurements. The results can be considered good when the mean deviation is 10^{-5} K, if it rises to 10^{-3} K means that results are not good and the noise is very high. If the values are within the right range the background shown in the software is white, if they are slightly out of range it is yellow, if they are far out of range it is red (figure 3.9). These indicators allow to understand both whether the selected data are

correct and whether the parameters entered at the beginning are good or should be changed.

Numeric Results					
Probing Depth	Temperature In...	Temperature D...	Total to Charac...	Total Temperat...	Time Correctior
6.31 mm	0.0184 K	-	0.242	0.146 K	0.1000 s
6.29 mm	0.0139 K	-	0.241	0.132 K	0.1000 s
0.860 mm	0.0293 K	-	0.00450	0.110 K	4.450e-015 s
3.99 mm	0.00892 K	-	0.0971	0.479 K	0.0984 s
6.34 mm	0.0178 K	-	0.245	0.875 K	0.1000 s
6.74 mm	0.0287 K	-	0.277	0.988 K	0.1000 s

Figure 3.9 - Example of warnings

Even if the TPS is a very precise method, the experiments can be repeated, to estimate the precision in the recording. The pause between measurements can be estimated, as the same space has to be recreated as the first measurement. Firstly the extension of the temperature distribution can be estimated when the recording has finished. The diameter of the temperature distribution is:

$$D = 6 (k \cdot t)^{\frac{1}{2}} \quad \text{Eq. 3.8}$$

that is more or less three times the probing depth. In order to obtain the time to the temperature to go back to isothermal conditions the inverse formula of the one just written here can be used. So this time is the square of the diameter divided by the thermal diffusivity that is the same value of 36 times the transient recording. For a metal sample, fifteen minutes is enough to equalize the temperature. After every new experiment the *Output of Power* and the *Measuring Time* have to be put again. So briefly the experiment consists of putting the sensor between two specimens of the same material and creating an isothermal space. After that the temperature can be increased and the measurement of some data can be taken. The software gives us the charts, some data that will be analyzed, and after that the thermal properties of the material will be calculated. If the experiment is to be repeated the machine needs to wait at least fifteen minutes to equalize the temperature of the sensor.

4. EXPERIMENTAL WORK

The aim of this project deals with the study of the thermal properties of Al-Si alloys. Samples studied are based on aluminium with a silicon content of 9%. The samples studied have a cylindrical shape: they have a diameter of 7.5 cm and a height of 4.5 mm. Three different pairs of samples are tested: one with a polished and an unpolished side and two with a coated and an uncoated (unpolished) side. There are two different types of coating: one is thinner, it is only 15-20 μm (figure 4.3) and the other one is 130-150 μm (figure 4.4). The coating is made by Polyethylene Oxide, PEO, which is a water-soluble thermoplastic material. This material is used in various fields due to its characteristics such as in the production of adhesives, paper, inks, ceramics, electronics and of course, as in this case, for coatings.



Figure 4.1 - Unpolished side



Figure 4.2 - Polished side

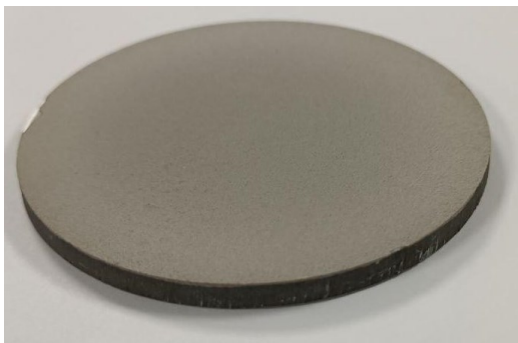


Figure 4.3 – Coated side (15-20 μm)



Figure 4.4 – Coated side (130 – 150 μm)

In order to reach the best results, several experiments are carried out using different methods. The measurements have been done as described in the chapter 3.3: the sensor is put between the two specimens; to insulate the specimens a little bit more,

two bricks on the top and above them are used, and then everything is firmly clamped together. The specimens are closed under a dome and then the software is used to set the parameters. In order to find the most suitable and promising method to evaluate the thermal properties, different modules are applied.

4.1 Test

4.1.1 Isotropic Method

Firstly, the Isotropic Method is used for the specimen with one surface polished and the other unpolished. This is the simplest processing, requiring only a few input data. Initial measurements are taken with the parameters described in table 4.1.

Table 4.1 - Parameters of the Isotropic Method

<i>Temperature</i>	21 °C
<i>Radius</i>	6,403 mm
<i>TCR</i>	0,004693 K ⁻¹
<i>Disk Type</i>	Kapton 5501
<i>Available Probing Depth</i>	4,00 mm
<i>Insulation</i>	Brick
<i>Sensor resistance</i>	6,679079 Ω

To decide on suitable time and power pairs, random combinations are chosen at the beginning in order to see what the results are. In particular, high times and low power are used initially. The output power and the measuring time combinations used are reported in table 4.2.

Table 4.2 - Output power/measuring time combinations for the polished side of the uncoated sample with the Isotropic Method

Output Power [W]	0,1	0,3	0,5	0,6	0,8	0,85	0,9
Measuring Time [s]	5 - 10 - 20	5	5	2 - 5 - 10	2	5	2 - 5
Output Power [W]	1	1,2	1,3	1,4	1,5	1,6	2
Measuring Time [s]	1 - 2 - 4 - 5 - 10	3	3 - 4 - 5	1 - 3 - 4	1 - 2 - 4 - 5 - 10	4	1 - 2 - 3 - 4 - 5 - 10

The first line of each table shows the power that is used, while the second line shows the times used to take measurements with that power. After having tried using low power and high measuring times, and seeing that the results obtained are not satisfactory, it is tried to raise the power and to lower the time. However, again the results obtained with these combinations are not satisfactory, in fact in the literature [29, 30] the values of the thermal conductivity of aluminium alloys are around 160 W/mK. In this case the average values of the thermal properties obtained are such as thermal conductivity about 42 W/mK for the polished surface and 43 W/mK for the unpolished one, thermal diffusivity and specific heat with values reported in table 4.3.

Table 4.3 - Thermal properties with the Isotropic Method for the sample uncoated

	<i>Polished surface</i>	<i>Unpolished surface</i>
Thermal Conductivity	42,06 W/mK	43,23 W/mK
Thermal Diffusivity	5,164 mm ² /s	4,48 mm ² /s
Specific Heat	8,16 MJ/m ³ K	9,66 MJ/m ³ K

For the selected set of parameters the Isotropic Method is not suitable.

4.1.2 Slab Method for uncoated side of specimens

Afterwards, another method is tested, characterised by radially propagation of heat into the sample and it is called the Slab Method. The first sample tested is the one also used in the previous method, the initial parameters being those given in table 4.4. These parameters will be used for all the specimens studied with the Slab Method.

Table 4.4 - Parameters of the Slab Method

Temperature	21 °C
Radius	6,403 mm
TCR	0,004693 K ⁻¹
Disk Type	Kapton 5501
Thickness of Slabs	4,5 mm
Available Radial Probing Depth	4,00 mm
Insulation	Brick
Sensor resistance	6,679079 Ω

There are two additional parameters here compared to the Isotropic Method, which are the *Thickness of Slabs* and the *Available Probing Depth*, which in this case is radial, because heat is no longer applied perpendicularly to the surface. The polished surface is the first side analysed. As with the other method, the choice of combinations begins with a range of both time and power, the time having a range from 1 s to 4 s, and the power form 0,1 W to 1,5 W, as it is described in table 4.5.

Table 4.5 - Output power/measuring time combinations for the polished side of the uncoated sample with the Slab Method

Output Power [W]	0,1	0,3	0,5	0,6	0,8	1	1,2	1,5
Measuring Time [s]	1	1 - 2 - 3 - 4	1	1 - 3	1	1	1	1

After choosing the combinations a chart is fundamental to calculate the thermal properties: the *Transient Chart*, that shows the increase of sensor temperature with

the time. The curve in this chart must be continuous and always increase. The range of points for calculating the thermal properties has to be chosen in this graph. In the figure 4.5, all the combinations tested are shown.

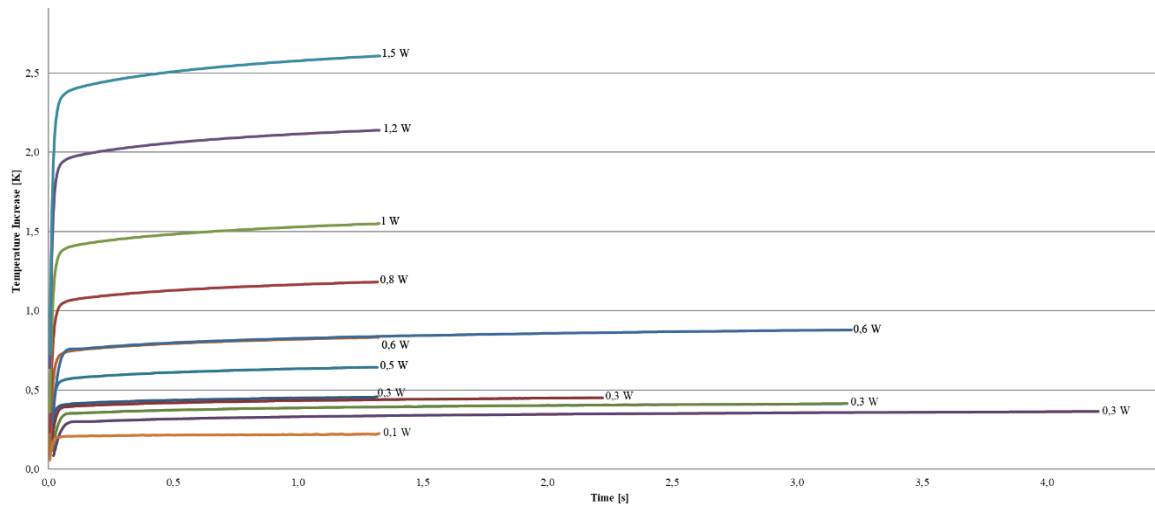


Figure 4.5 - Transient Chart of the polished side of the uncoated sample with the Slab Method

In order to understand whether the choice of experimental data set for analyzing the thermal properties of Al-Silicon alloys is suitable, four other parameters must be studied: probing depth, temperature increase, the ratio between total and the temperature increase and the ratio between total time for the measurement and the characteristic time. They are shown in the figure 4.6 - 4.9; and to elucidate the reported data in all charts the green columns are the considered ones. The first parameter to be considered is the *Probing Depth* (figure 4.6). It represents how much the heat propagates in the specimens. In the Slab Method the propagations of the heat is radial, so the limit is a little bit less than the diameter of the specimens, for this reason the maximum value of this parameter is set to 35 mm. All the found experimental data show lower values with respect to 35 mm, except for the combinations with an output power equal to 0,1 W.

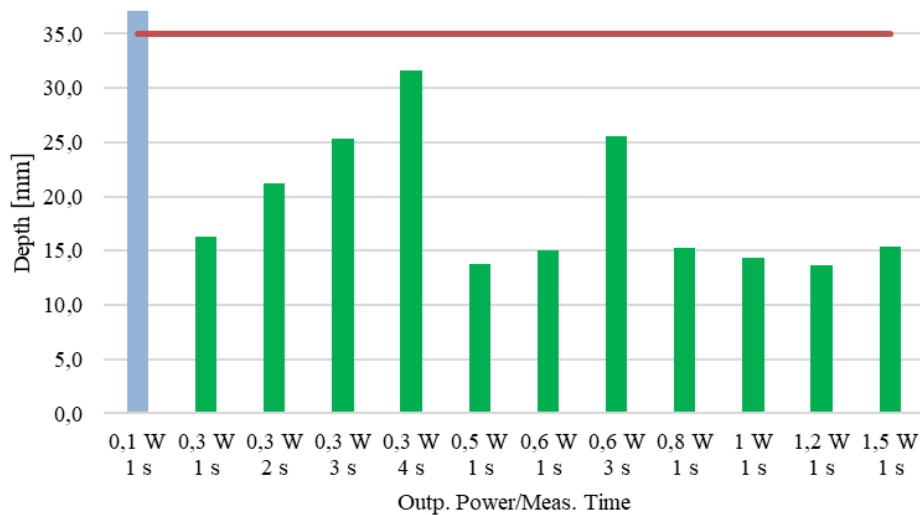


Figure 4.6 - Probing Depth of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)

The second parameter is the *Temperature Increase* that describes how much temperature increases between the first selected point and the last one. Usually the temperature has to grow at least 0,05 K. This parameter is reported in the figure 4.7; it shows that only the measurements with power higher than 0.8 W increase the temperature in the right range.

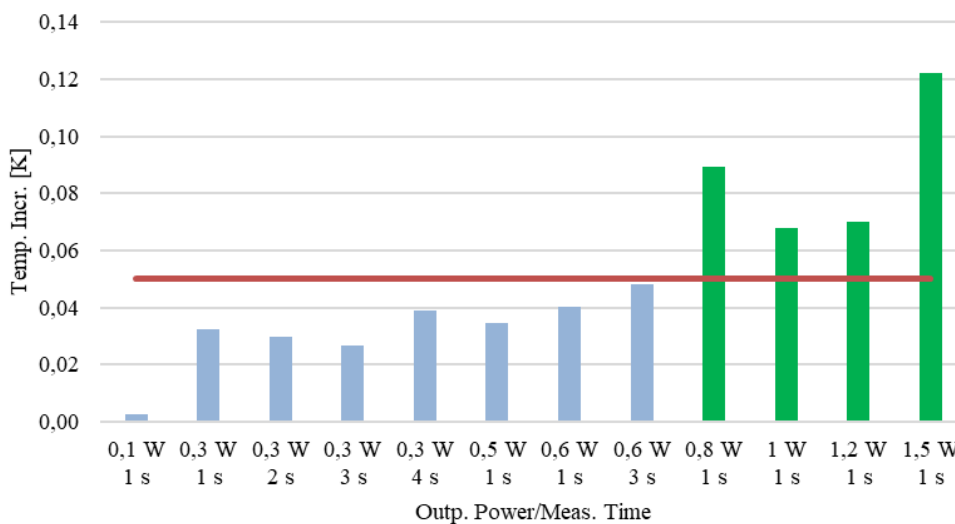


Figure 4.7 - Temperature Increase of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)

The *ratio between Total and the Temperature Increase* describes the total temperature increment, so it corresponds to the temperature value of the last sample.

It depends on the measurements, but usually this ratio has to be higher than 1,25 K. As it is represented in the figure 4.8 the adequate parameters are the last three, so when the output power is higher than 1 W.

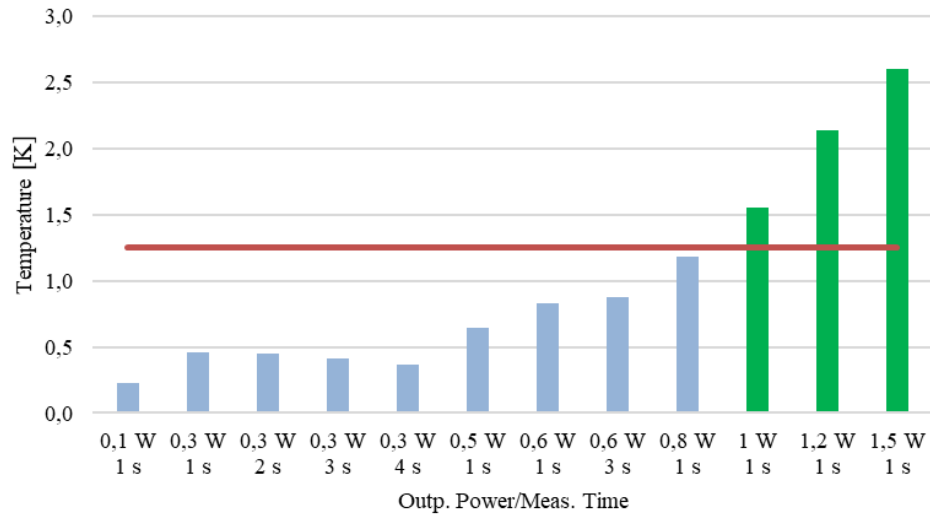


Figure 4.8 - Total/Temp. Incr. of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)

The last parameter that is important for the analysis is the *ratio between Total Time for the measurement and the Characteristic Time*. It has a range from 0,33 to 1. All the combinations produce useless results (figure 4.9). The data are only a little bit higher than the range. It needs a measurement time that has to be shorter, but 1 s is already the smallest usable time.

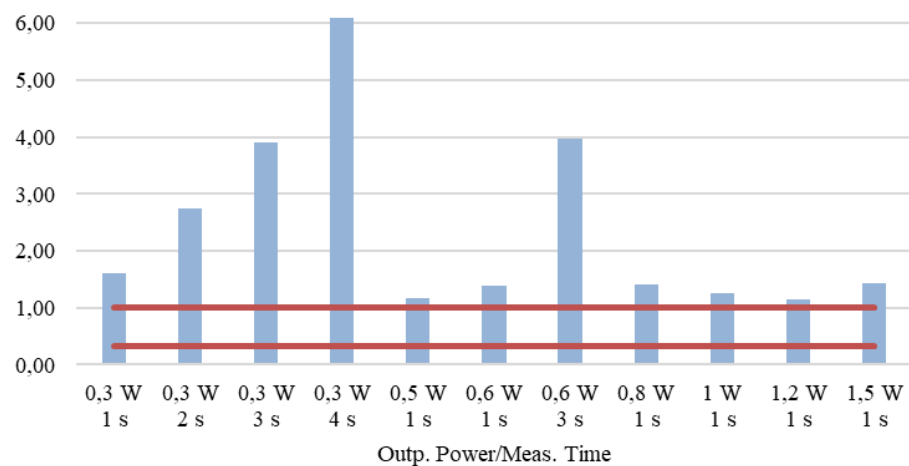


Figure 4.9 - Total/Characteristic Time of the polished side of the uncoated sample with the Slab Method (green columns are acceptable)

From these graphs it is clear that combinations with high power and low time are the ones that are in the range for the most of the parameters.

A graph that the software returns after choosing the data is the *Residual Chart*, which shows the difference that there is between the measurement data and the fitted data. This chart has to be random, as it happens in the case of this specimen (figure 4.10). In this graph only those combinations that seemed correct by the previous analysis, are selected.

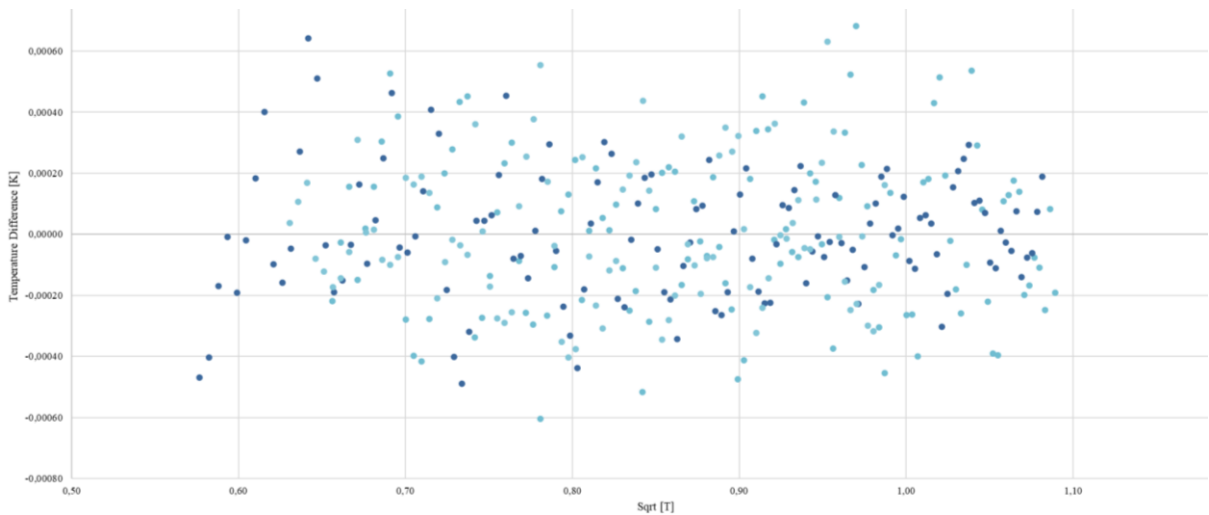


Figure 4.10 - Residual Chart of the polished side of the uncoated sample with the Slab Method

After choosing the data in the transient graph, analysing the four parameters and the residual graph for the polished surface, the best combinations are the ones with an output power in a range from 1 W to 1,5 W and a measuring time equal to 1 s.

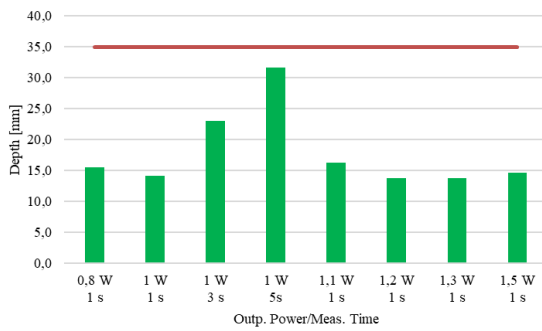
The Slab Method is used for the surface of the specimens that are uncoated. For all the specimens the four parameters are studied, from them the right combinations of power and time are found out.

For the unpolished surface of the uncoated specimens, the combinations of power and time, shown in the table 4.6, are used. The power used has a range from 0,8 W to 1,5 W, but this choice is not random. In fact, the starting point of the experiments are the values of the polished side of the same specimen.

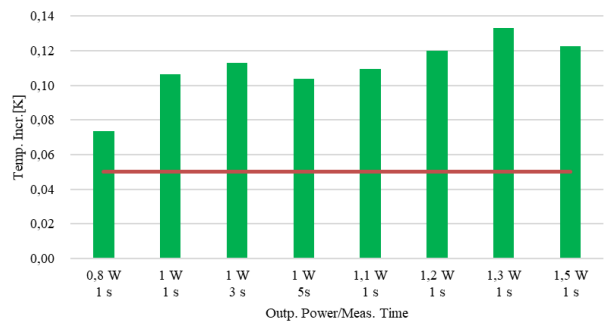
Table 4.6 Output power/measuring time combinations for the unpolished side of the uncoated sample with the Slab Method

Output Power [W]	0,8	1	1,1	1,2	1,3	1,5
Measuring Time [s]	1	1 - 3 - 5	1	1	1	1

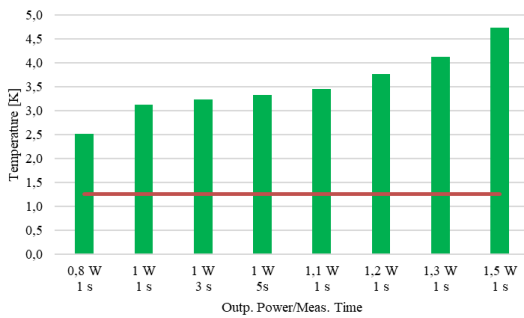
The four parameters are analysed and they are shown in the figure 4.11. From this, all the combinations are good, and all the results are inside the range, except for one parameter, the ratio between the total and the characteristic time (figure 4.11.d). As for the polished side it needs a measurement time that has to be little, but 1 s is already the smallest usable time.



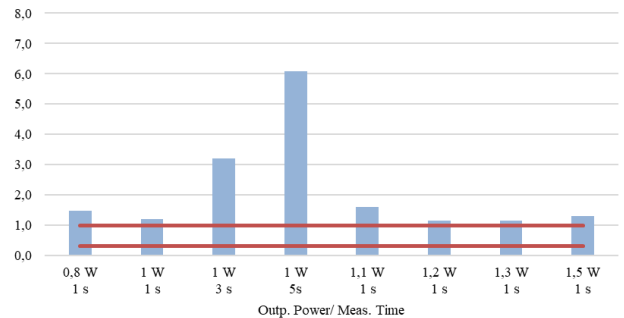
a - Probing Depth



b - Temperature increase



c - Total/Temp.Incr.



d - Total/Characteristic Time

Figure 4.11 - Parameters of the unpolished side of the uncoated sample with the Slab Method (green columns are acceptable)

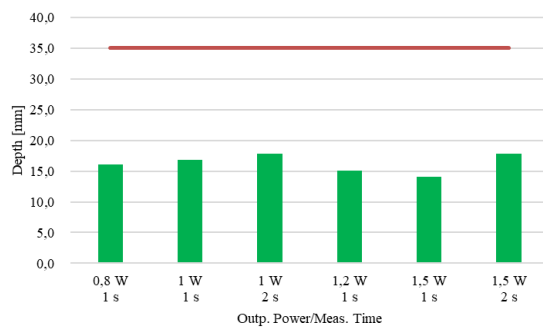
Considering all the experimental parameters explained before, the best combinations for the unpolished side are power in a range from 0,8 W and 1,5 W and a time from 1 s to 5 s (higher than 1 s only for some powers). So for this uncoated specimen the combinations of the output power and the measuring time, that give reasonable results, are very similar from one side to the other. This means that having a polished surface does not affect the thermal properties.

The uncoated surface of the specimens with the other surface coated by 15 - 20 μm of PEO is studied next. The combinations used are very similar to the ones used for the other specimens (table 4.7), because the samples have surfaces of the same nature. The range of the output power is from 0,8 W and 1,5 W, and the measuring time used is from 1 s to 2 s.

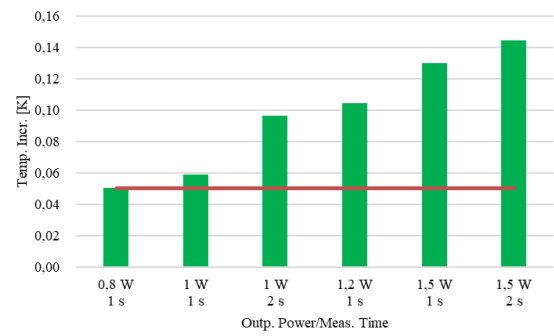
Table 4.7 - Output power/measuring time combinations for the uncoated side of the coated sample (15-20 μm) with the Slab Method

Output Power [W]	0,8	1	1,2	1,5
Measuring Time [s]	1	1 - 2	1	1 - 2

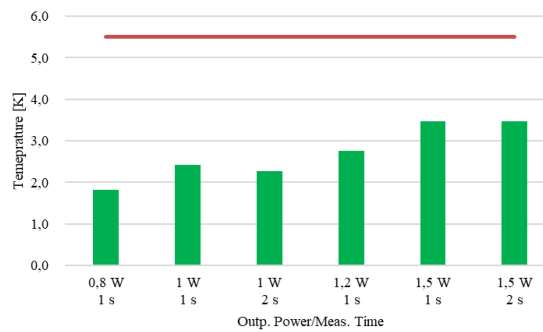
All combinations result between the ranges for three parameters: the probing depth (figure 4.12.a), the temperature increase (figure 4.12.b and the ratio between the total and the temperature increase (figure 4.12.c). As for the other uncoated surfaces the ratio between the total and the characteristic time is out of the range (figure 4.12.d).



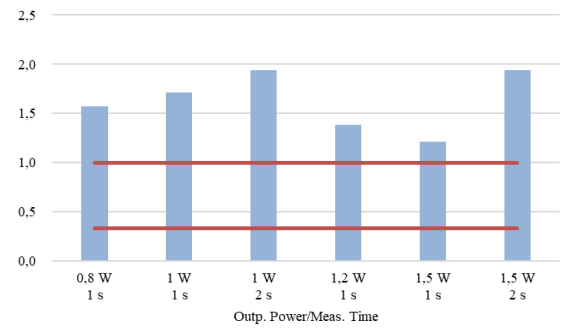
a - Probing Depth



b - Temperature Increase



c - Total/Temp.Incr



d - Total/Characteristic Time

Figure 4.12 - Parameters of the uncoated side of the coated sample (15 - 20 μm) with the Slab Method (green columns are acceptable)

The fourth surface to be analyzed with this method is the uncoated side of the sample with the thicker coating (130 - 150 μm). The combinations of power and time used are reported in the table 4.8. Also in this case, the initial values are not chosen randomly, but they are similar to the combinations used for the previous sample as the two samples have surfaces of the same nature.

Table 4.8 - Output power/measuring time combinations for the uncoated side of the coated sample (130-150 μm) with the Slab Method

Output Power [W]	0,5	0,8	0,9	1	1,2	1,3	1,6	2
Measuring Time [s]	1	1 - 2 - 5	1 - 2 - 3 - 5	1 - 2 - 5	1	1	1	1

The figure 4.13 represents the parameters that are fundamental to choose the right combinations.

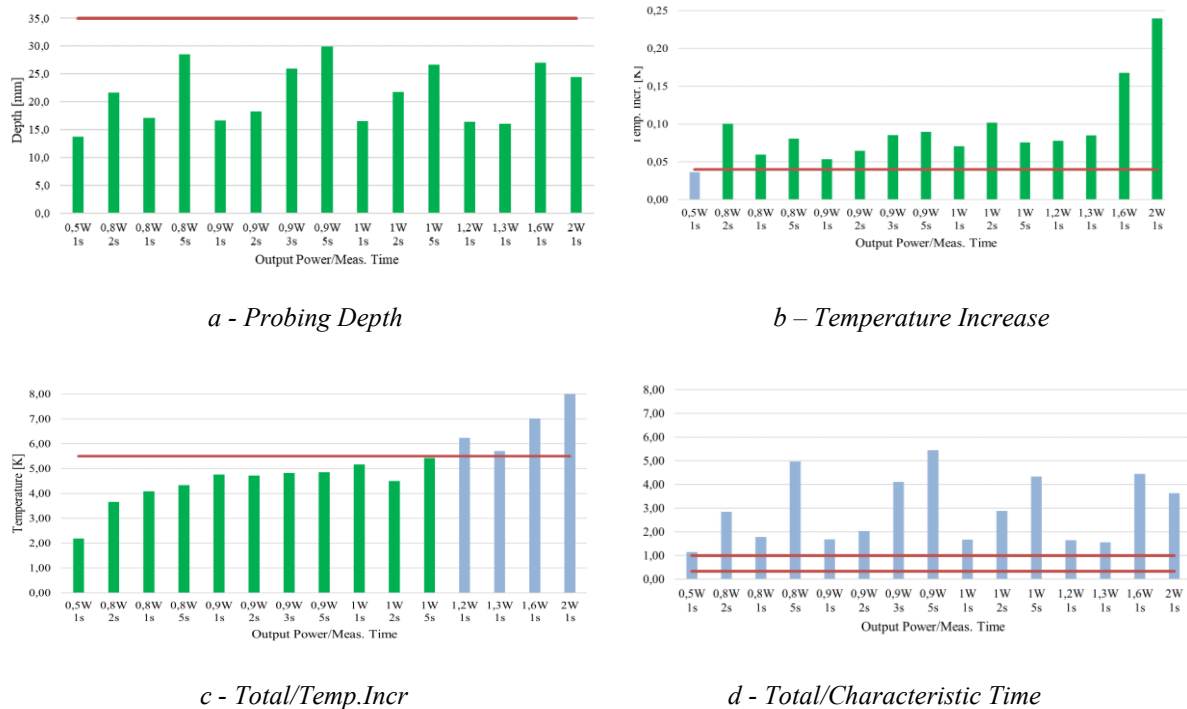


Figure 4.13 - Parameters of the uncoated side of the coated sample (130 - 150 μ m) with the Slab Method (green columns are acceptable)

When the power is about 0,5 W, the temperature remains constant (figure 4.13.b); on the other hand, using a power greater than or equal to 1.3 W, the temperature of the last sample is too high and therefore outside the allowed limits (figure 4.13.c). Also in this case the ratio between the total and the characteristic time is out of range, but as for the other analysis there 1 s is already the smallest usable time. From the parameter graphs obtained, it is therefore noted that the ideal power is a range of 0,8 W to 1 W and a measuring time of 1 s to 5 s.

4.1.3 Thin Film Method for coated side of specimens

For the coated surface of the two specimens the Thin Film Method is used. It is utilised to evaluate the thermal conductivity of thin insulating films or coatings on conducting substrates of bulk or slab type. The measurement process involves calculating the thermal resistance over the film, which allows to determinate the thermal conductivity while taking into account the film thickness. The initial parameters are reported in table 4.9.

Table 4.9 - Parameters of the Thin Film Method

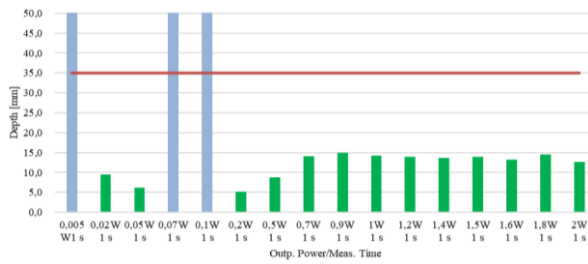
<i>Temperature</i>	21 °C
<i>Area of the sensor</i>	129 mm ²
<i>Radius of the sensor</i>	6,403 mm
<i>Thickness of the sensor</i>	25 µm
<i>Thermal Conductivity of the sensor</i>	35,63 mW/mK
<i>TCR</i>	0,004693 K ⁻¹
<i>Film Thickness</i>	20 µm
<i>Background material</i>	Al alloy
<i>Disk Type</i>	Kapton 5501
<i>Sensor resistance</i>	6,679079 Ω

The *Film Thickness* is 20 µm only because the first specimen analysed is the one with the thinner coating equal to 15 - 20 µm. At the beginning different combinations are used, which are shown in the table 4.10. The starting point is a very low power, unlike the other surfaces studied, as the behaviour of this surface is unknown. The measurement time is similar to that used in the other methods and it is always 1 s for this sample.

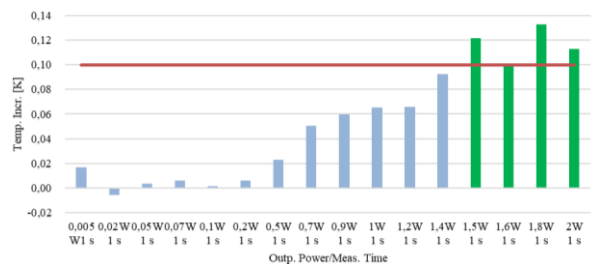
Table 4.10 Output power/measuring time combinations for the coated side of the coated sample (15-20 μm) with the Thin Film Method

Outp. Power [W]	0,05	0,02	0,05	0,07	0,1	0,2	0,5	0,7
Measuring Time [s]	1	1	1	1	1	1	1	1
Outp. Power [W]	0,9	1	1,2	1,4	1,5	1,6	1,8	2
Measuring Time [s]	1	1	1	1	1	1	1	1

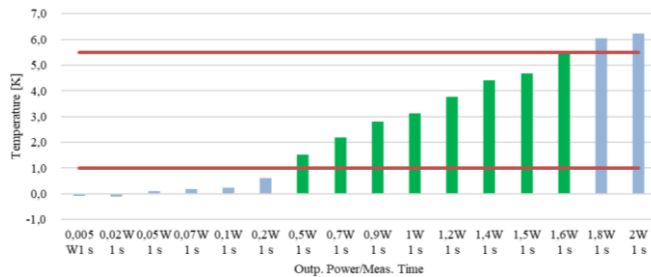
To find the most suitable combinations, the four parameters are analysed also with this method.



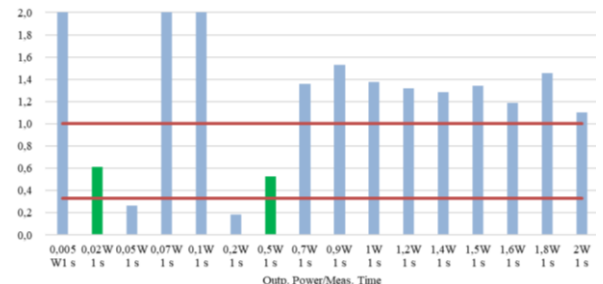
a - Probing Depth



b - Temperature Increase



c - Total/Temp.Incr



d - Total/Characteristic Time

Figure 4.14 - Parameters of the coated side of the coated sample (15 - 20 μm) with the Thin Film Method (green columns are acceptable)

If the probing depth is considered, all the combinations with the power higher than 0,2 W are in the right range (figure 4.14.a). Instead, the values of the temperature increase are in the range only for high power such as 1,5 W (figure 4.14.b). The values of the ratio between the total and the temperature incremental are in the range for a value of output power from 0,5 W to 1,5 W (figure 4.14.c). The ratio between the total and the characteristic time is in the range only for power equal to 0,02 W and 0,5 W (figure 4.14.d). Without considering the last parameter as in the study of the other samples, as the values not in the range are not too far off, it would seem that the appropriate power is 1.5 W. However, looking at the values of the thermal properties of all combinations (table 4.11), they are different from each other. For very low powers, the values of thermal conductivity are negative, which means that heat moves from a body at a lower temperature to a body at a higher one, when it should be the other way around.

Table 4.11 - Thermal properties of the coated side of the coated sample (15-20 μm) with the Thin Film Method

Outp.power.	Meas.time	Th.Conductivity	B Th. Conductivity	B Th. Diffusivity	B Spec.Heat
0,005W	1s	-0,00904 W/mK	-2,48 W/mK	4454,863 mm ² /s	-0,001 MJ/m ³ K
0,02W	1s	-0,00950 W/mK	575,27 W/mK	17,012 mm ² /s	33,816 MJ/m ³ K
0,1W	1s	-0,06549 W/mK	60,10 W/mK	8,762 mm ² /s	6,859 MJ/m ³ K
0,1W	1s	-0,12956 W/mK	282,03 W/mK	5756,248 mm ² /s	0,049 MJ/m ³ K
0,1W	1s	-0,09218 W/mK	254,69 W/mK	3766,639 mm ² /s	0,068 MJ/m ³ K
0,2W	1s	0,60131 W/mK	68,94 W/mK	5,255 mm ² /s	13,120 MJ/m ³ K
0,5W	1s	0,41179 W/mK	104,03 W/mK	16,756 mm ² /s	6,209 MJ/m ³ K
0,7W	1s	0,34116 W/mK	148,14 W/mK	45,513 mm ² /s	3,255 MJ/m ³ K
0,9W	1s	0,31735 W/mK	155,55 W/mK	52,779 mm ² /s	2,947 MJ/m ³ K
1W	1s	0,31619 W/mK	145,01 W/mK	42,382 mm ² /s	3,422 MJ/m ³ K
1,2W	1s	0,30016 W/mK	146,69 W/mK	43,714 mm ² /s	3,356 MJ/m ³ K
1,4W	1s	0,28953 W/mK	147,30 W/mK	44,570 mm ² /s	3,305 MJ/m ³ K
1,5W	1s	0,32015 W/mK	149,93 W/mK	46,883 mm ² /s	3,198 MJ/m ³ K
1,6W	1s	0,14659 W/mK	146,86 W/mK	46,824 mm ² /s	3,136 MJ/m ³ K
1,8W	1s	0,16018 W/mK	145,34 W/mK	44,656 mm ² /s	3,255 MJ/m ³ K
2W	1s	0,31115 W/mK	147,77 W/mK	44,088 mm ² /s	3,352 MJ/m ³ K

*B = Background

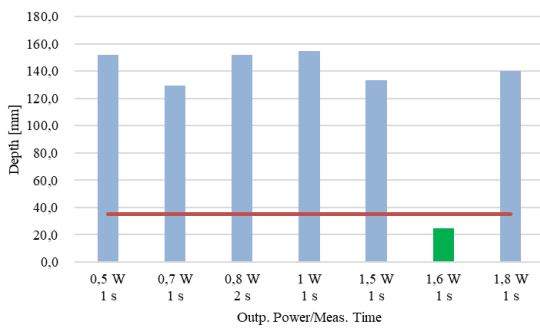
So, the Thin Film Method is not the right method to calculate the thermal properties for this specimen. This method is used also for the coated surface of the other sample. In this case the coating is thicker, because it is 130 - 150 μm , so in the initial parameters on the *Film Thickness* the value is 150 μm . As for the other methods

different combinations of output power and measuring time are used, as it is shown in the table 4.12.

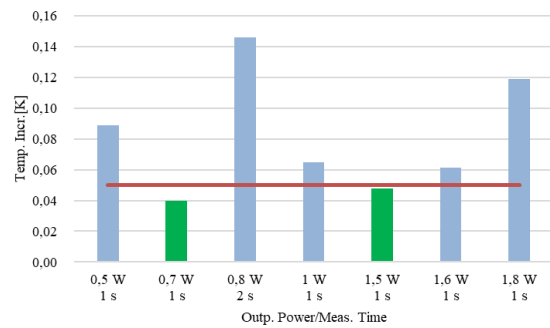
Table 4.12 Output power/measuring time combinations for the coated side of the coated sample (130-150 μm) with the Thin Film Method

Outp. Power [W]	0,5	0,7	0,8	1	1,5	1,6	1,8
Measuring Time [s]	1	1	2	1	1	1	1

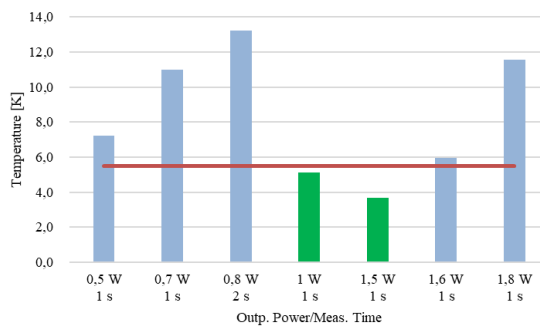
From the analysis of the parameters, it is difficult to detect the best combination power/time because in all four of them a different couple fits better.



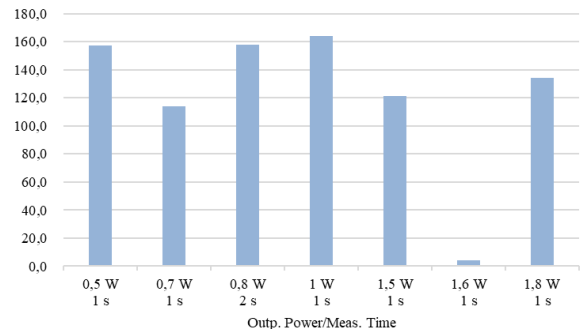
a - Probing Depth



b - Temperature Increase



c - Total/Temp.Incr.



d - Total/Characteristic Time

Figure 4.15 - Parameters of the coated side of the coated sample (130 - 150 μm) with the Thin Film Method (green columns are acceptable)

In the analysis of the probing depth (figure 4.15.a) the only couple that could be right is the one with the power equal to 1,6 W and a measuring time of 1 s. While for temperature increase analysis, the best combinations are formed by an output power of 0,7 W or 1,5 W and measuring time of 1 s (figure 4.15.b). The ratio of total and temperature increase, on the other hand, has data in the range only for combinations with a power of 1 W and 1,5 W and a time of 1 s (figure 4.15.c). Consequently, a more suitable pair cannot be found, which gives satisfactory thermal property results. This method is not the most indicated for calculating the thermal properties of this sample, too.

4.1.4 Slab Method for coated side of specimens

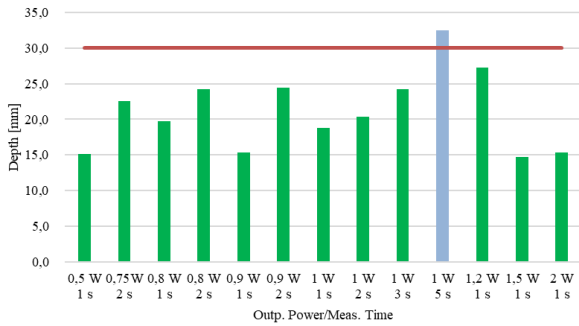
For this reason the Slab Method is also used for the part coated of these two samples. The initial parameters used are the same as for the uncoated surfaces. For the specimens with the thinner coating (15 - 20 μm) the combinations of output power and measuring time are reported in the table 4.13.

Table 4.13 - Output power/measuring time combinations for the coated side of the coated sample (15-20 μm) with the Slab Method

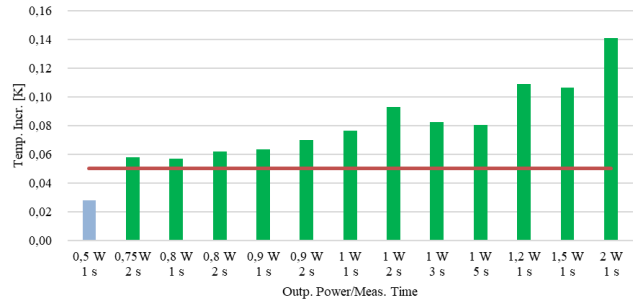
Outp. Power [W]	0,5	0,75	0,8	0,9	1	1,2	1,5	2
Measuring Time [s]	1	2	1 - 2	1 - 2	1 - 2 - 3 - 5	1	1	1

From the analysis of the four considered parameters, the combination with an output power equal to 1 W and a measuring time equal to 5 s is out of range when probing depth is considered (figure 4.16.a). From the analysis of the temperature increase (figure 4.16.b) only the combination with the output power equal to 0,5 W is a little bit lower than the range. Considering the ratio between the total and the temperature increase the combinations with the power higher than 1,5 W are out of range (figure

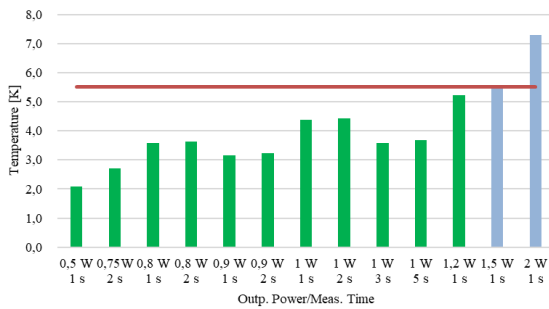
4.16.c). As for all the other specimens the ratio between total and the characteristic time is a little bit higher than the range (figure 4.16.d).



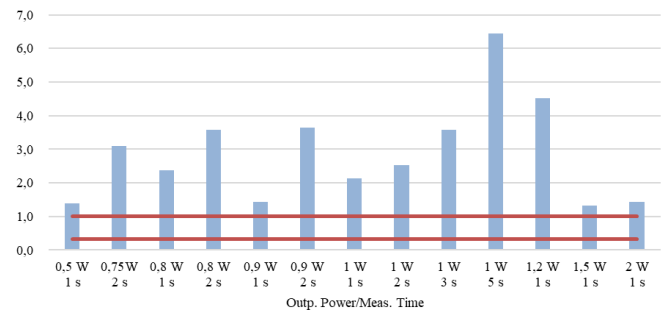
a - Probing Depth



b - Temperature Increase



c - Total/Temp.Incr.



d - Total/Characteristic Time

Figure 4.16 - Parameters of the coated side of the coated sample (15 - 20 μm) with the Slab Method (green columns are acceptable)

So, the best combinations are the ones with an output power in a range from 0,75 W and 1 W and a measurement time from 1 s to 3 s. Even if the combination 1,2 W and 1 s has the parameters in the range, it is not considered because its thermal diffusivity is too high (yellow column of figure 4.17).

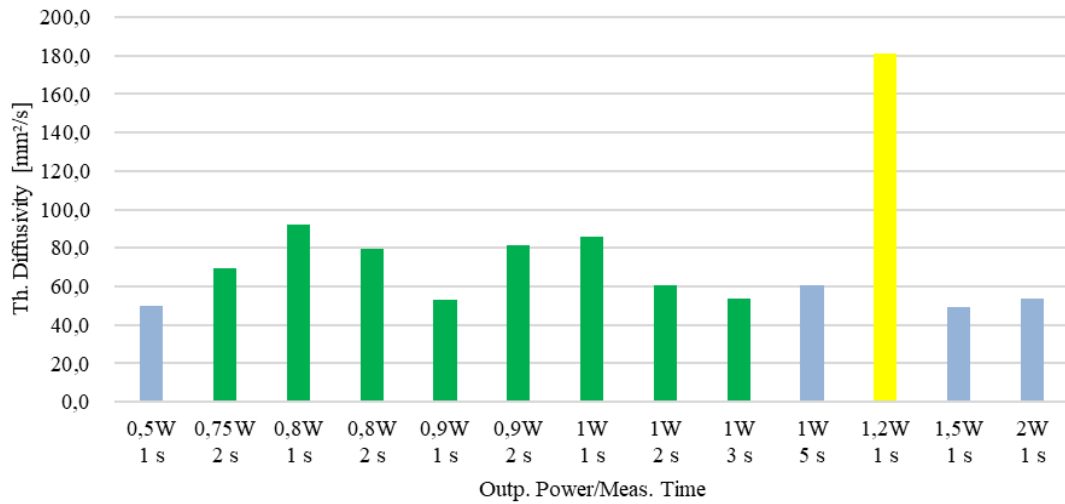


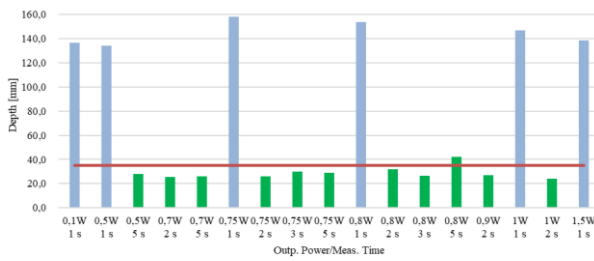
Figure 4.17 - Thermal Diffusivity of the coated side of the coated sample (15-20 μm) with the Slab Method

For the specimen with the thicker coating (130-150 μm) the combinations used are reported in the table 4.14.

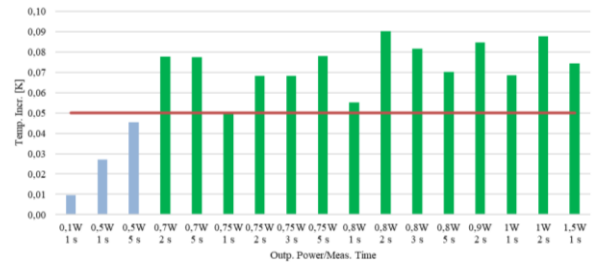
Table 4.14 - Output power/measuring time combinations for the coated side of the coated sample (130-150 μm) with the Slab Method

Output Power [W]	0,1	0,5	0,7	0,75	0,8	0,9	1	1,5
Measuring Time [s]	1	1 - 5	2 - 5	1 - 2 - 3 - 5	1 - 2 - 3 - 5	2	1 - 2	1

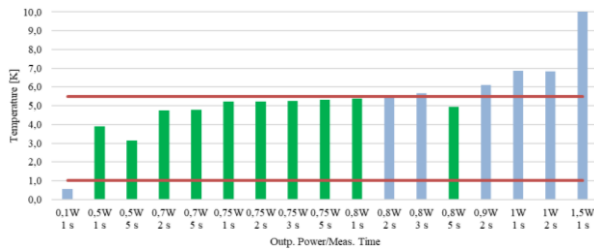
In the figure 4.18 the bar charts of the four parameters are shown.



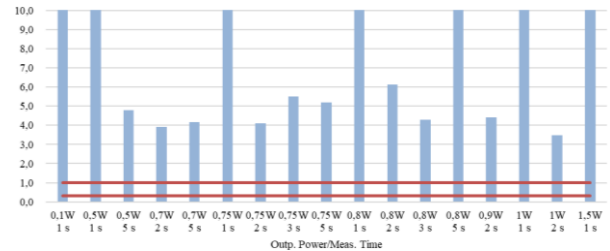
a - Probing Depth



b - Temperature Increase



c - Total/Temperature Increase



d - Total/Characteristic Time

Figure 4.18 - Parameter of the coated side of the coated sample (130 - 150 μm) with the Slab Method (green columns are acceptable)

For this case the measuring time is the parameter to focus on in order to decide on the appropriate combinations studying the probing depth (figure 4.18.a). In fact all the combinations with a time higher than 1 s have value within the range. On the other hand for the temperature increase only combinations with a power higher than 0,7 W can be considered, because they are in the range (figure 4.18.b). The ratio of total and the temperature increase shows that only the combinations with a power from 0,5 W to 0,8 W have value in the range (figure 4.18.c). As always, the ratio of total to characteristic time presents all values out of range (figure 4.18.d). By cross-referencing the study of the four parameters, the best combinations are obtained to calculate the thermal properties: the output power is in a range from 0,7 W to 0,75 W and the measuring time has to be higher than 1 s.

4.2 Parameters

After these analysis the choice of output power and measuring time that give satisfactory results are reported in the table 4.15.

Table 4.15 - Summary of satisfactory parameters

	<i>Output Power [W]</i>	<i>Measuring Time [s]</i>
<i>Isotropic Method</i>		
Polished side of the uncoated sample	Not found	Not found
Unpolished side of the uncoated sample	Not found	Not found
<i>Slab Method</i>		
Polished side of the uncoated sample	1 - 1,5	1
Unpolished side of the uncoated sample	0,8 - 1,5	1 - 5
Uncoated side of the coated sample (15 - 20 μ m)	0,8 - 1,5	1 - 2
Coated side of the coated sample (15 - 20 μ m)	0,75 - 1	1 - 3
Uncoated side of the coated sample (130 - 150 μ m)	0,8 - 1	1 - 5
Coated side of the coated sample (130 - 150 μ m)	0,7 - 0,75	2 - 5
<i>Thin Film Method</i>		
Coated side of the coated sample (15 - 20 μ m)	Not found	Not found
Coated side of the coated sample (130 - 150 μ m)	Not found	Not found

From the data reported in table 4.15, the Slab Method is the most consistent method for obtaining satisfactory results. The values for power and time are more or less all the same. For the uncoated surfaces the output power is always in a range from 0,8 W to 1,5 W and a time near 1 s. For the coated surface the output power is a little bit smaller, it is near 0,75 W and a time a little bit higher. The other two methods have no values that give acceptable results. In conclusion, stating that whether the surface is polished or unpolished has no effect on the choice of combinations of output power and measuring time for the calculation of thermal properties, whereas if it is coated, the range of combinations changes slightly.

5. DATA ANALYSIS

In this chapter, thermal properties such as thermal conductivity, thermal diffusivity and specific heat are described for each sample analysed. The data of these properties are calculated using the output power and the measuring time ranges defined in chapter 4. However, to compare how these properties change with increasing power, the time used has to be the same. For the uncoated sample and the coated one with the coating equal to 15 - 20 μm the measuring time considered is 1 s. The third sample with the thicker coating proved to be a different case, therefore it is analyzed last. In this project work, to understand the thermal properties data that the software provides, the thermal circuit can be compared to an electrical circuit. This means that the flow of heat through a thermal resistor is entirely analogous to the flow of current through an electrical resistor. So the equation describing the behaviour of one system can be transformed into the equation of the other system by simply changing the symbols of the variables. For example in the heat power equation:

$$q = \frac{\Delta T}{R_T} \quad \text{Eq 5.1}$$

if the symbol of the thermal potential ΔT is replaced by the symbol of the electric potential ΔV , the difference of potential, and the symbol for the thermal resistance R_T is replaced by the symbol for the electrical resistance R_E , the equation of current flow is obtained:

$$I = \frac{\Delta V}{R_E} \quad \text{Eq 5.2}$$

It is Ohm's law. The samples studied can thus be compared to resistors in an electrical circuit. In the case of the uncoated surface, there is only one resistor as the properties are calculated on a specimen that is all made of the same material. Consequently, as in circuits the electric current passes completely through the resistance according to Ohm's law (figure 5.1), the heat is passed through the sample. The thermal properties, computed in this situation, are taken as a reference for comparison with the results of the thermal properties of the coated surfaces measured.

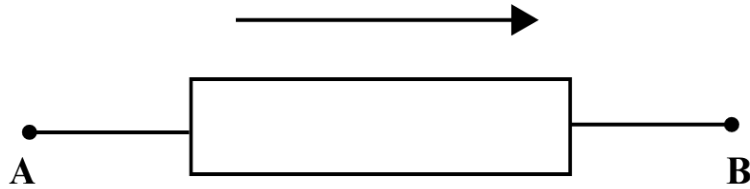


Figure 5.1 - Single resistor

On the other hand, in the coated side, two resistors are considered: one represented by the PEO coating and the other represented by the base. The system can be seen, by analogy, as a parallel connection of two resistors in which the thermal power is replaced by the current circulating in the two branches of the circuit. In this case then the heat generated by the passage of current through the sensor is divided between the two parts of the sample (figure 5.2.)

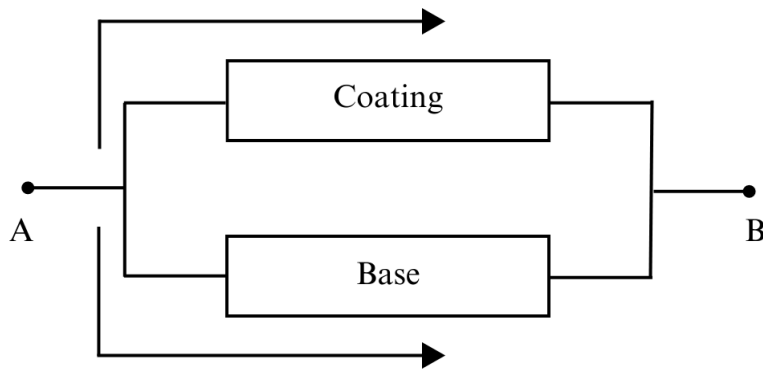


Figure 5.2 - Parallel connection of two resistors

When there are two resistors in parallel, the equivalent resistance is calculated according to the formula:

$$\frac{1}{R_{eq}} = \frac{1}{R_1} + \frac{1}{R_2} \quad \text{Eq. 5.3}$$

The reciprocal of the equivalent resistance is equal to the sum of the reciprocals of the individual resistances. Knowing that the resistance is equal to:

$$R = \frac{L}{k \cdot A} = \frac{L}{k \cdot L \cdot h} = \frac{1}{k \cdot h} \quad \text{Eq. 5.4}$$

where k is the thermal conductivity, A is the area of the resistor formed by L , length of the resistor, and h , height of the resistor; the formula can be turned into:

$$\frac{1}{R_{\Sigma}} = \frac{1}{R_{coat}} + \frac{1}{R_{base}} \quad Eq. 5.5$$

$$k_{\Sigma} \cdot h_{\Sigma} = k_{coat} \cdot h_{coat} + k_{base} \cdot h_{base} \quad Eq. 5.6$$

where λ_{Σ} represents the thermal conductivity of the entire sample, λ_{coat} and λ_{base} are the thermal conductivity of the part with the coating and the base respectively, h_{Σ} is the total height of the sample, h_{coat} is the height of the part with the coating and h_{base} is the height of the base. However, the heat is not only radially transmitted through the two parts of the sample, but is also transferred between the two parts: heat therefore passes from the coating part to the base part. In order to know how deep the properties are actually calculated in the sample, the equation 5.6 has to be slightly modified, as the whole height of the base is not involved, but only a fraction of it. The equation then becomes:

$$k_{\Sigma} \cdot h_{\Sigma} = k_{coat} \cdot h_{coat} + k_{base} \cdot h_{base} \cdot x \quad Eq. 5.7$$

in which x is:

$$x = \frac{h_{base\ eff.}}{h_{base}} \quad Eq. 5.8$$

where $h_{base\ eff.}$ is the effective height up to where the heat reaches. According to what was just described, the results of the properties are shown below.

5.1 Uncoated sample

The first specimen studied is the uncoated one. In the table 5.1 the results of the three properties obtained are reported. The measuring time considered is equal to 1 s for every measurement.

Table 5.1 - Thermal properties of the uncoated sample

	Output Power [W]	Th. Conductivity [W/mK]	Th. Diffusivity [mm²/s]	Specific Heat [MJ/m³K]
Polished side	1	154,06	43,16	3,57
	1,2	159,59	47,95	3,33
	1,5	159,59	50,42	3,17
Unpolished side	0,8	161,97	52,03	3,11
	1	168,02	42,62	3,94
	1,1	168,86	50,21	3,75
	1,2	168,93	42,99	3,93
	1,3	165,44	40,53	4,08
	1,5	166,51	45,88	3,63

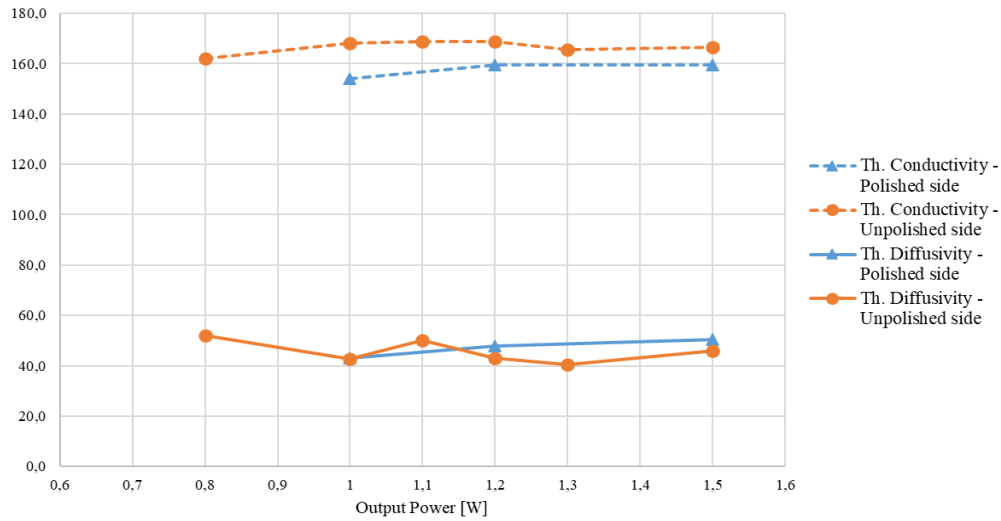


Figure 5.3 - Thermal Conductivity and Thermal Diffusivity of the uncoated sample

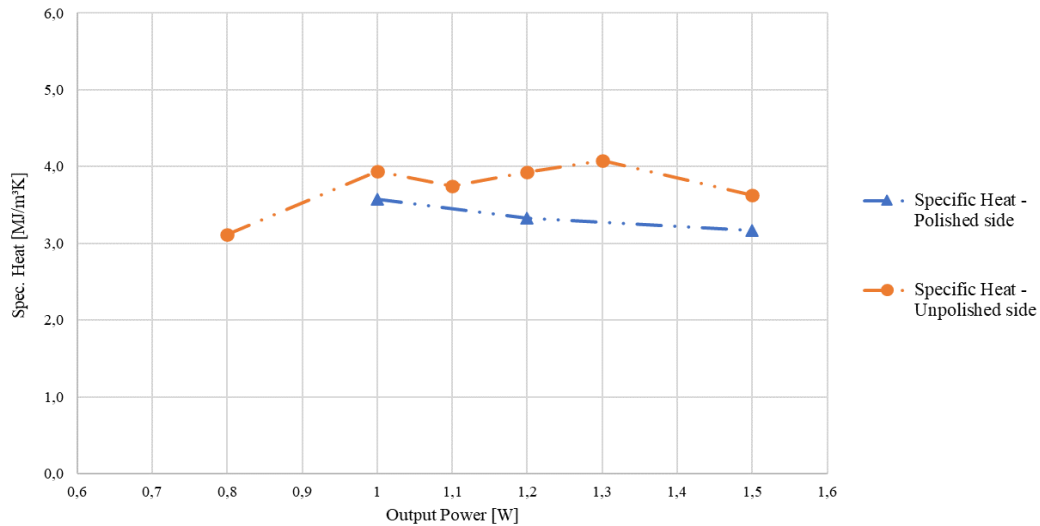


Figure 5.4 - Specific Heat of the uncoated sample

From the figures 5.3 and 5.4 the values of the three properties obtained are reported as the power increases; it can be seen that the trend of the properties is independent of power, because the values are all very similar.

5.2 Coated sample (15-20 μm)

The second specimen studied is the coated sample with the thinner coating (15 - 20 μm) . As for the previous sample, to compare the value of the thermal properties obtained (table 5.2), the measuring time used is equal to 1 s.

Table 5.2 - Thermal properties of the uncoated sample

	<i>Output Power [W]</i>	<i>Th. Conductivity [W/mK]</i>	<i>Th. Diffusivity [mm²/s]</i>	<i>Specific Heat [MJ/m³K]</i>
<i>Coated side</i>	0,8	189,90	92,39	2,06
	0,9	179,18	85,35	2,23
	1	180,50	85,91	2,10
<i>Uncoated side</i>	0,8	177,79	62,84	2,83
	1	179,04	74,12	2,42
	1,2	168,47	51,01	3,30
	1,5	169,60	44,75	3,79

According to the introduction to this chapter, the thermal conductivity found is the sum of thermal conductivity of the coating and the base (multiplied by the respective heights). However, from the figure 5.5 it can be seen that comparing the values obtained from the specimen with the coating and the values of the uncoated specimen, taken as a reference, they are very similar.

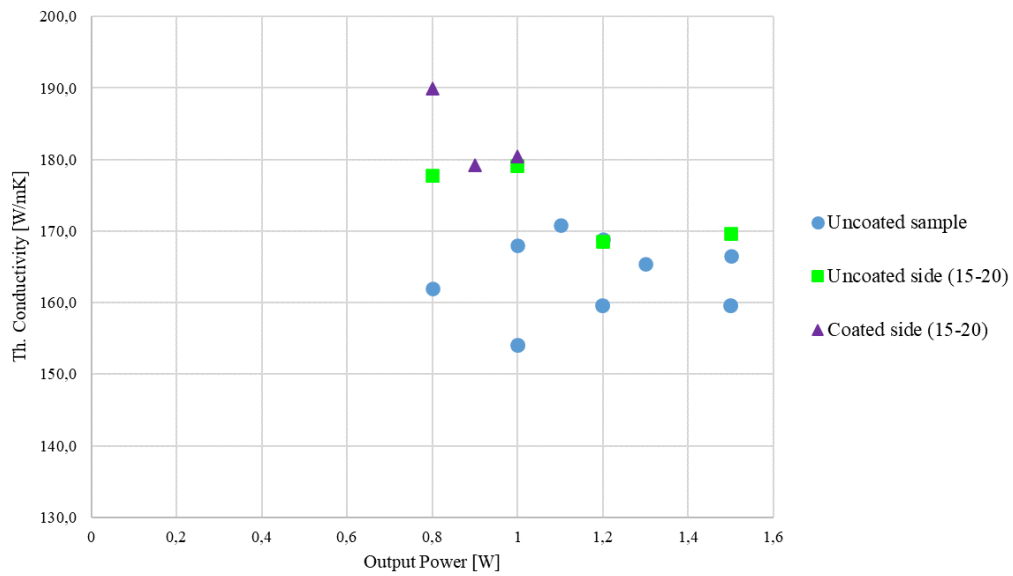


Figure 5.5 - Comparison of Thermal Conductivity of coated and uncoated sample

Consequently, it can be stated that the proposed formula (eq 5.6) doesn't work with such a thin coating. The analysis then proceeds in the same way as for the uncoated sample. In the figure 5.6 and 5.7 it is described how the properties change as the power increases.

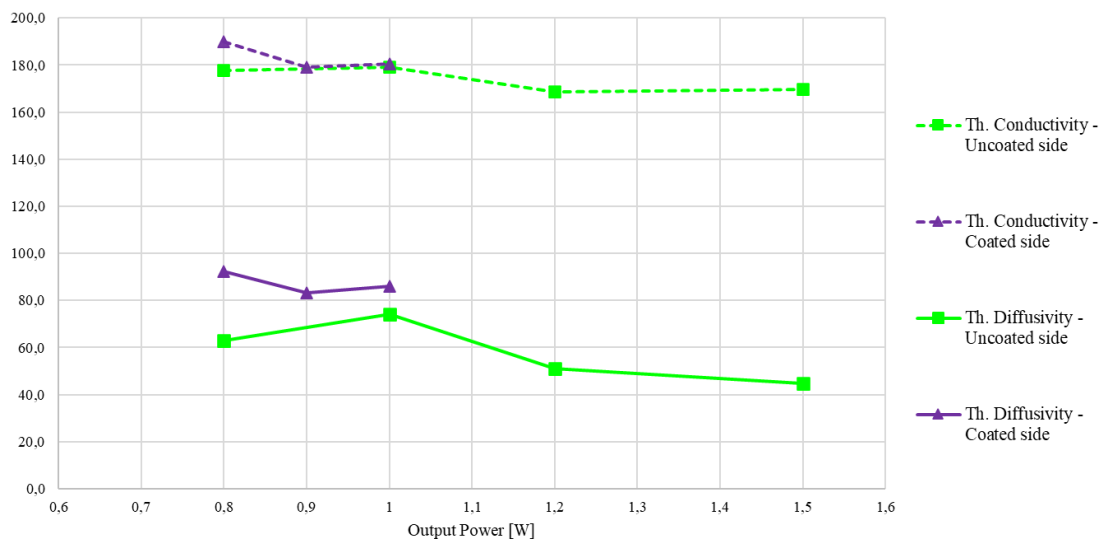


Figure 5.6 - Thermal Conductivity and Thermal Diffusivity of the coated sample (15-20 μm)

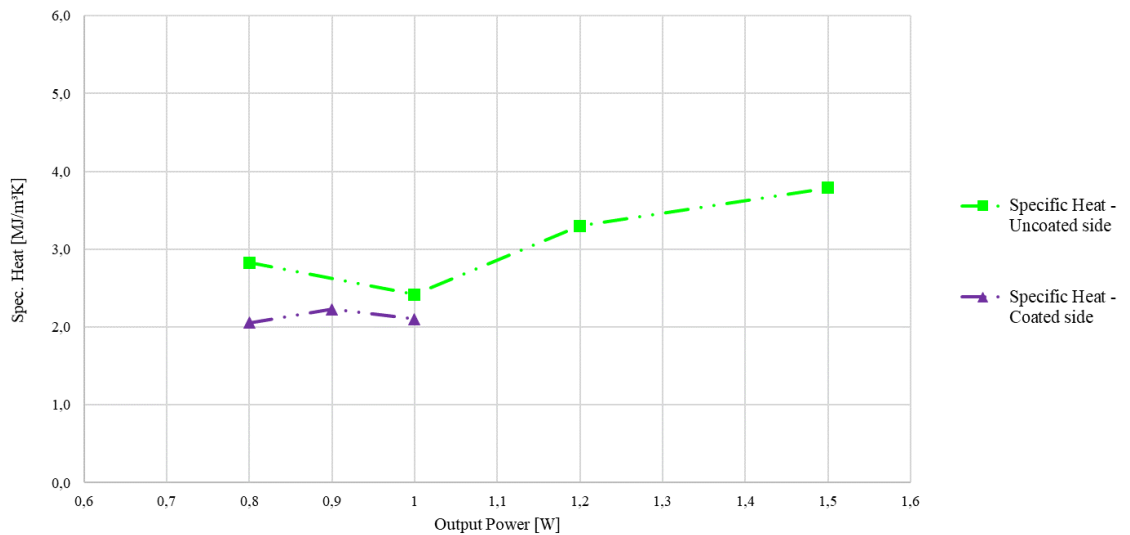


Figure 5.7 - Specific Heat of the coated sample (15-20 μm)

In the figure 5.6 it is shown that the thermal properties of the coated side have a higher value than the uncoated one. The thermal conductivity of the coating is usually lower than the uncoated side, the reason why in this case it is higher can not be explained.

5.2 Coated sample (130-150 μm)

Several measurements are made for this sample before satisfactory results are obtained. According to the previous chapter, the time range for the coated side is from 2 to 5 s, while for the uncoated side it is from 1 to 5 s. The power that gives satisfactory results for the coated side is from 0,7 to 0,75 W, while for the uncoated side it is from 0,8 to 1 W. For this sample, the two surfaces are analysed separately. Firstly the coated side is studied. Table 5.3 reports the properties values using a measuring time equal to 5 s.

Table 5.3 - Thermal properties of the coated side of the coated sample (130-150 μm)

	<i>Output Power</i> [W]	<i>Th. Conductivity</i> [W/mK]	<i>Th. Diffusivity</i> [mm ² /s]	<i>Specific Heat</i> [MJ/m ³ K]
<i>Coated side</i>	0,7	140,46	51,09	2,75
	0,75	142,17	56,92	2,50
<i>Average value</i>		<i>141,31</i>		

Thermal conductivity of this side is compared to the thermal conductivity of the uncoated sample in the figure 5.8.

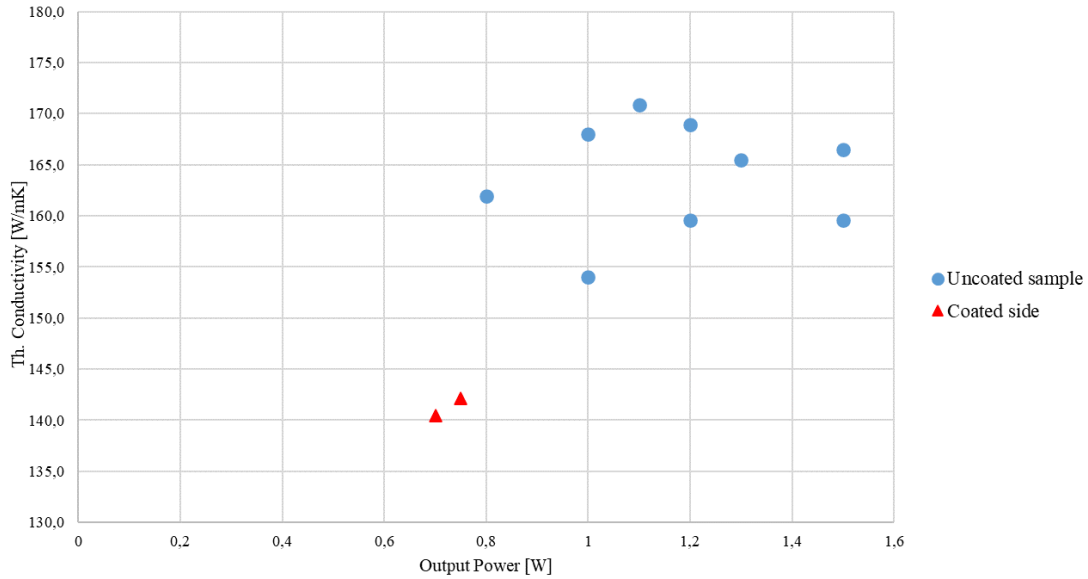


Figure 5.8 - Comparison of thermal conductivities

From the data and the figure 5.8 it is clear that the measurements taken by the coated side have a lower thermal conductivity, also the output power applied is lower, than the other. By applying the law of the parallel resistor (equation 5.7), the value of the thermal conductivity of the coating can be found:

$$k_{\Sigma} = \frac{k_{coat} \cdot h_{coat} + k_{base} \cdot h_{base} \cdot x}{h_{\Sigma}} \quad Eq 5.9$$

Where $k_{\Sigma}=141,31$ W/mK, $h_{\Sigma}= 4,5$ mm, $h_{coat}=130$ μ m, $k_{base}= 163,89$ W/mK, $h_{base} = h_{\Sigma} - h_{coat} = 4,5 - 0,13 = 4,37$ mm, and $x = 100\%$. For the values of the thermal conductivity the average value of the thermal conductivity of the coated side of this sample and the average value of the thermal conductivity of the uncoated sample are used respectively. Using these values, the thermal conductivity of the coating is:

$$k_{coat} = \frac{k_{\Sigma} \cdot h_{\Sigma} - k_{base} \cdot h_{base} \cdot x}{h_{coat}} = \frac{141,31 \cdot 4,5 - 163,89 \cdot (4,5 - 0,130) \cdot 1}{0,130} = - 617,72 \text{ W/mK}$$

The result obtained is negative and therefore not acceptable. It is thus necessary to change some values in the formula. Many are fixed such as the average value of thermal conductivities and the coating thickness. The value that can be changed, however, is the height at which the total thermal conductivity is calculated. The percentage of h_{base} involved is represented by x . If it is decreased, the values of the thermal conductivity of the coating change according to the table 5.4

Table 5.4 - Changes of k_{coat} as x varies

x [%]	100	90	88,4	88,35	88,3	80
k_{coat} [W/mK]	-617,72	-66,80	21,34	24,10	26,85	484,12

$x = 88,35$ % is the fraction of the height of the base to which the thermal conductivity has been actually calculated. The calculated average thermal conductivity of the uncoated side is approximately 164 W/mK. According to the table 5.4 the thermal conductivity of the coating is approximately 24 W/mK. Therefore, to obtain the thermal conductivity calculated by the TPS machine, the value of the coating (24 W/mK) is subtracted from the average value. This means that the thermal conductivity calculated by the TPS machine is approximately 140 W/mK:

$$164 \text{ W/mK} - 24 \text{ W/mK} = 140 \text{ W/mK}$$

It is indicatively the average value obtained in the table 5.3, therefore the values of thermal conductivity obtained are satisfactory.

In the figure 5.9 it is shown how the three properties change with the increasing power.

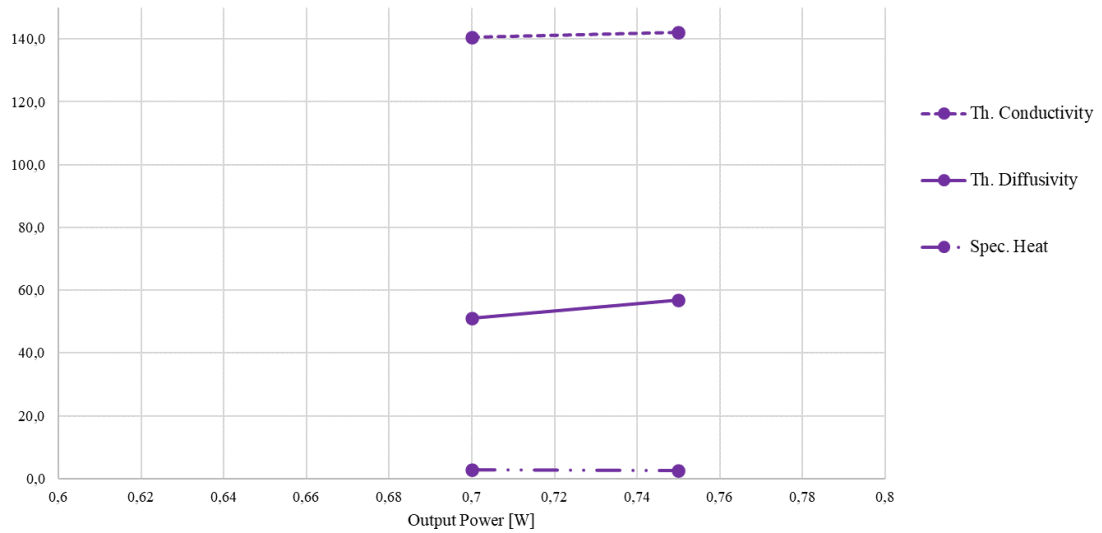


Figure 5.9 - Thermal properties of the coated side of the coated sample (130-150 μm)

For the uncoated side, the measuring time values have a range from 1 s to 5 s. At first, a measuring time of 1 s is considered, which is the time used for all other uncoated surfaces. The results obtained are reported in table 5.5.

Table 5.5 - Thermal properties of the uncoated side of the coated sample (130-150 μm)

	Output Power [W]	Th. Conductivity [W/mK]	Th. Diffusivity [mm²/s]	Specific Heat [MJ/m³K]
Uncoated side	0,8	167,55	139,96	1,20
	0,9	160,78	125,17	1,28
	1	168,20	175,31	0,96

The thermal conductivity is very similar to the other ones calculated for the other uncoated sides. However, the thermal diffusivity and the specific heat are different from those expected. In the figures 5.10 And 5.11 these values are compared to the value obtained in the uncoated specimen.

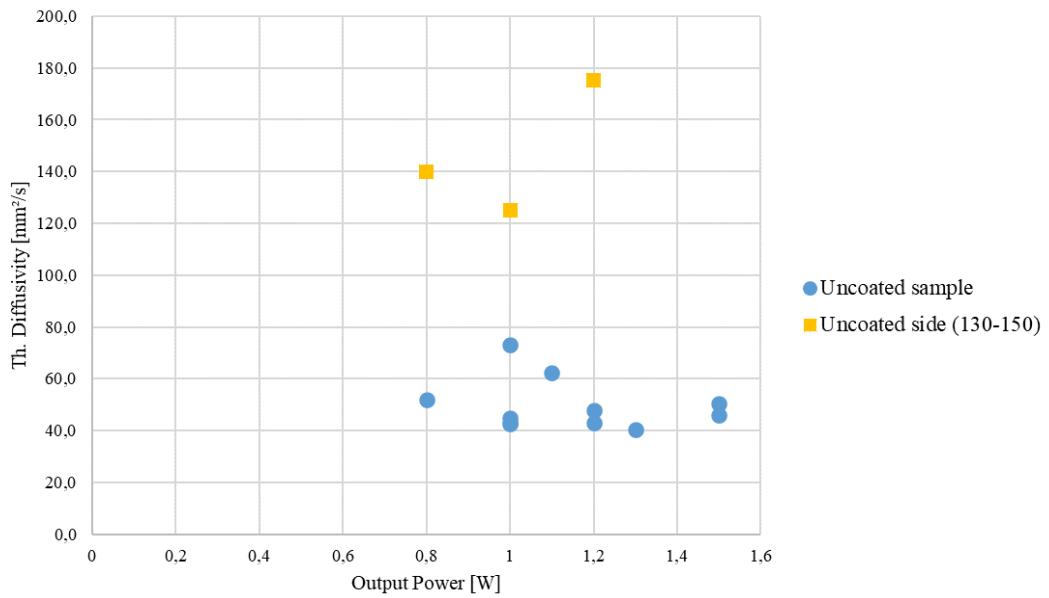


Figure 5.10 - Comparison of Thermal Diffusivity of the uncoated side of the coated sample (130-150 μm) with the uncoated sample

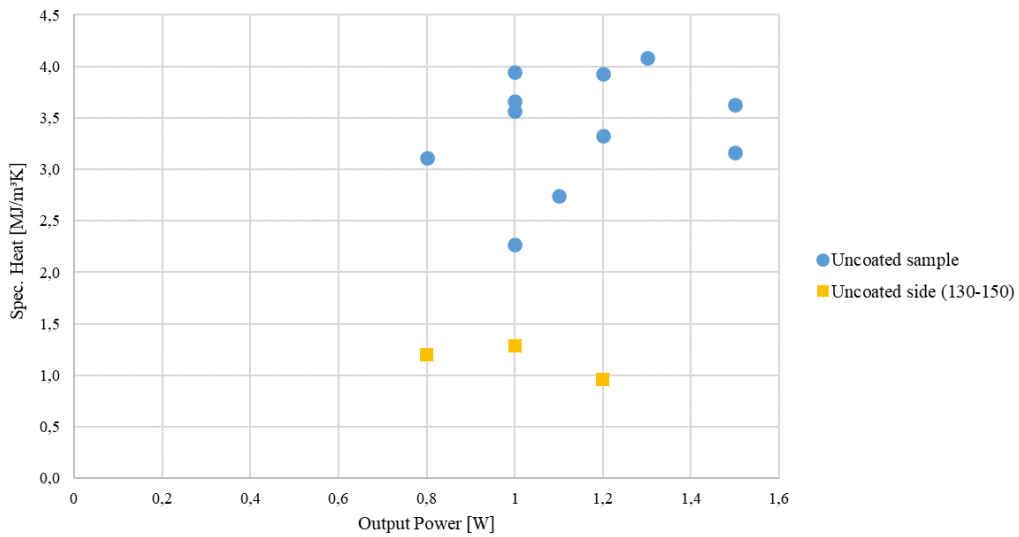


Figure 5.11 - Comparison of Specific Heat of the uncoated side of the coated sample (130-150 μm) with the uncoated sample

It is clear that they are not the expected values. However, the data obtained depends on the choice of points in the transient graph. By choosing other points, the values obtained are shown in table 5.6.

Table 5.6 - Thermal properties of the uncoated side of the coated sample (130-150 μm) -
Measuring Time = 1s

	Output Power [W]	Th. Conductivity [W/mK]	Th. Diffusivity [mm²/s]	Specific Heat [MJ/m³K]
Uncoated side	0,8	133,98	69,20	1,94
	0,9	130,84	58,58	2,23
	1	133,67	64,35	2,08

The values of the last two properties are close to the average values of the other samples, however, the thermal conductivity value is lower than the expected ones. With this combination of time and power, satisfactory results are not obtained. A measuring time that gives satisfactory results in terms of thermal diffusivity and specific heat is 5 s (table 5.7).

Table 5.7 - Thermal properties of the uncoated side of the coated sample (130-150 μm) -
Measuring Time = 5s

	Output Power [W]	Th. Conductivity [W/mK]	Th. Diffusivity [mm²/s]	Specific Heat [MJ/m³K]
Uncoated side	0,8	135,02	48,83	2,77
	0,9	134,53	53,59	2,51
	1	136,41	54,26	2,51

Another measuring time that gives satisfactory results in terms of specific heat and thermal diffusivity is 2 s. Before considering the values obtained, the four parameters that are used to find suitable combinations must be reviewed, in particular, the value of the ratio between total time for the measurement and the characteristic time. The value should be between 0,33 a 1. The values of this ratio are always out of the range, nevertheless, the combination of power and time that should be chosen is the one with the smallest errors.

Table 5.8 - Total/Characteristic Time

Total/Char. Time	Output Power [W]	Meas. Time = 2 s	Meas. Time = 5 s
	0,8	2,85	4,96
	0,9	2,04	5,45
	1	2,88	4,33

From the table 5.8, that reports the value of the ratio that has to be considered, the measuring time that is better to use is equal to 2 s. The results obtained using this measuring time are reported in the table 5.9.

Table 5.9 - Thermal properties of the uncoated side of the coated sample (130-150 μm) - Measuring Time = 2s

	<i>Output Power [W]</i>	<i>Th. Conductivity [W/mK]</i>	<i>Th. Diffusivity [mm²/s]</i>	<i>Specific Heat [MJ/m³K]</i>
<i>Uncoated side</i>	0,8	145,04	57,08	2,54
	0,9	132,46	60,80	2,18
	1	143,95	57,71	2,49

The values of thermal diffusivity and specific heat are close to the desired values; but the values of thermal conductivity are not satisfactory, because they are lower than the average value expected. It can be concluded that a suitable measuring time and output power combination is not found for this specimen.

5.5 Comparison between found data and old data

The uncoated sample was studied 10 years ago. The machine used was one older than the one used today, but of the same type. However, the TPS method and the sensor material used are the same: the properties were taken in the radial direction using the Slab Method and the sensor used was made of Kapton. The results obtained are reported in the table 5.10.

Table 5.10 - Thermal properties of the uncoated sample (10 years ago)

	<i>Output Power [W]</i>	<i>Th. Conductivity [W/mK]</i>	<i>Th. Diffusivity [mm²/s]</i>	<i>Specific Heat [MJ/m³K]</i>
<i>Uncoated side</i>	4,5	142,7	56,0	2,551
	5	143,2	56,2	2,549
	5,5	139,5	55,8	2,500

These results are slightly different from those obtained now. It is common for results to differ when different machines or technologies are used in experiments.

This is because the performance and characteristics of the equipment can affect the accuracy and precision of the measurements. Then the size of the sensor used can also have an impact on the results obtained, but in the report which size of sensor was used isn't defined. Regarding the difference in power used, it is possible that newer technology requires less power to achieve the same results. However, the similarity in the trend of the data is clear, as it suggests that despite the differences in equipment, the fundamental relationship between power and values obtained is consistent: as ten years ago, also today the thermal conductivity and the thermal diffusivity are independent of the power (figure 5.12).

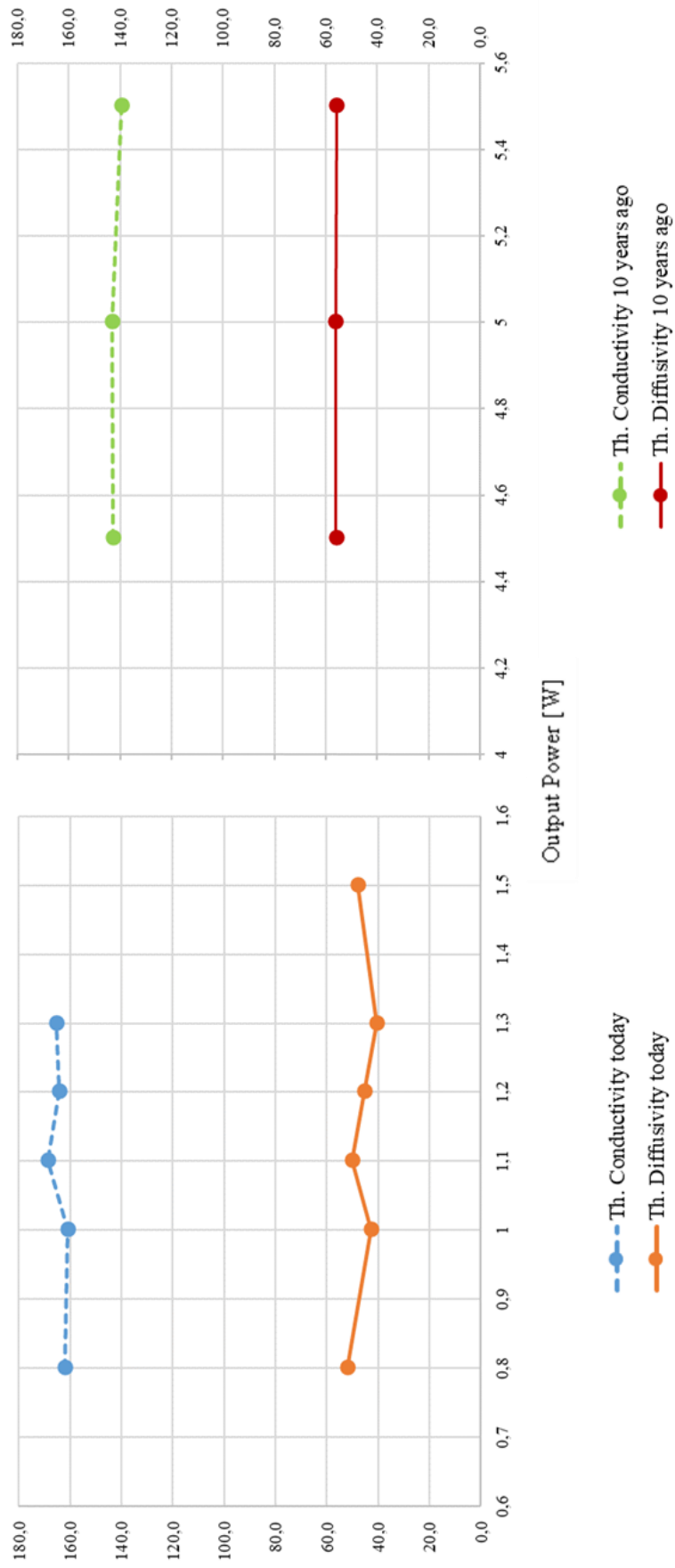


Figure 5.12 – Thermal Conductivity and Thermal Diffusivity of the incoated sample studied today and 10 years ago

5.6 Comparison between found data and literature data

In literature values of the thermal properties of 46000 aluminium alloys can be found. However, the data that can be compared are only those of the uncoated sample. The value that can be compared is the average between the value of the polished and the unpolished surface of the uncoated sample, as the thermal properties should not be affected by this process. Only the value of thermal conductivity is considered, all other thermal properties depend on it so they won't be analysed in this paragraph. Table 5.1 shows that the average value found for the polished surface is 158 W/mK and for the unpolished side it is 166 W/mK. It can therefore be affirmed that the average value found of this property for an EN 46000 alloy is approximately 162 W/mK. All the values of the thermal properties in this chapter are considered at room temperature because the tests with the Hot Disk Machine were carried out at 21°C. These values found are compared with three different sources whose values are slightly different.

Firstly, the data sheet of a 46000 alloy proposed by Raffmetal is studied. This company is the largest producer of recycled aluminium alloys in Europe and has studied and created data sheets for several alloys. The value given in the sheet for thermal conductivity is 110 - 120 W/mK, which is slightly lower than the value found. Then a software is used to calculate the thermal conductivity, specific heat and density of an alloy from its chemical composition. As input data, those in Table 5.11 are used (the chemical composition is the one found with the spectrometer).

Table 5.11 - Input: chemical composition

Elements	Al	Cu	Fe	Mg	Mn	Ni	Pb	Si	Ti	Zn
Chemical Composition	84,82	3,31	0,87	0,19	0,24	0,05	0,08	9,37	0,04	1,03

The value of thermal conductivity found is 106,37 W/mK. Even in this case, the value found is lower than the one found experimentally.

Lastly, according to an article written by Ozhoga-Maslovskaja et al. (2016), the thermal conductivity value for this type of alloy is 110 W/mK [31].

In all three different sources studied, the indicated thermal conductivity values are lower than those found experimentally.

CONCLUSIONS

The aim of this project is the study of the thermal properties of the aluminium alloy. The study of microstructure of the sample with the spectrometer and the microscopes show that the alloy of this final project is an EN 46000, an aluminium alloy with 9% of silicon, which have numerous applications in various industrial fields. The properties analysed are thermal conductivity, thermal diffusivity and specific heat. The properties were studied with the Hot Disk machine and the TPS method, which produce a series of very interesting results.

From the analysis of the three samples, the results achieved can be summarised as follows:

- For the uncoated side of the specimens two methods were tried. Firstly, the Isotropic Method is used but the values obtained were too low compared to what was expected. Then the Slab Method was used, in which the heat propagates radially through the sample. The latter is the best method applied to determinate the studied properties.
- Also for the coated side two methods were tested. The first one was the Thin Film Method, which would have been suitable precisely for the calculation of properties for thin layers. However, no suitable combinations of output power and measuring time were found. Therefore, the Slab method was also used for these sides, which returned property values similar to the expected values.

In conclusion the best method for analyzing these samples was the Slab Method. The results are reported in the table 1.

Table 6 - Obtained results from the samples under investigation

<i>Slab Method</i>	<i>Output power [W]; Meas. time [s]</i>	<i>Th. Conductivity [W/mK]</i>	<i>Th. Diffusivity [mm²/s]</i>	<i>Specific Heat [MJ/m³K]</i>
Polished side of the uncoated sample	1 - 1,5; 1	157,75	47,177	3,35
Unpolished side of the uncoated sample	0,8 - 1,5; 1	166,42	50,506	3,42
Coated side of the coated sample (130-150µm)	0,7 - 0,75; 5	142,59	72,144	2,08
Uncoated side of the coated sample (15-20µm)	0,8 - 1,5; 1	172,84	53,164	3,38
Coated side of the coated sample (15-20µm)	0,75 - 1; 1	177,48	72,05	2,55

It was not possible to calculate the parameters to obtain satisfactory data for the uncoated surface of the sample coated with a coating of 130-150 µm. In fact, based on the points chosen in the transient chart and the measuring time used, the results of the three properties show that: using a little time, only the thermal conductivity was good, but using a slightly higher time, the thermal conductivity decreased. Comparing these data with the ones obtained 10 years ago, although the equipment was different but the method used was the same (Slab Method), the thermal properties trend as power increases remains the same for an uncoated sample.

The study of this type of alloy is crucial for the future, as it is a material that will be used extensively from both primary production and waste. The possibility that by using suitable coatings its thermal properties will improve, it is not to be underestimated. The TPS method is also very innovative, as it is very precise, does not degrade the samples and allows many values to be obtained in a short time. On the other hand, finding the right combinations of output power and measuring time and the right method to study the material with, is not so immediate.

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