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Sina, Hossein; Iyengar, Srinivasan; Melin, Solveig

Published in:

Advances in powder metallurgy & particulate materials - 2012 : proceedings of the 2012 International Conference on Powder Metallurgy & Particulate Materials : PowderMet 2012, June 10-13, Nashville

2012

[Link to publication](#)

Citation for published version (APA):

Sina, H., Iyengar, S., & Melin, S. (2012). Ignition temperatures for Cu-Al and Ni-Al reactions in elemental powder mixtures using Differential Scanning Calorimetry. In I. Donaldson, & N. T. Mares (Eds.), *Advances in powder metallurgy & particulate materials - 2012 : proceedings of the 2012 International Conference on Powder Metallurgy & Particulate Materials : PowderMet 2012, June 10-13, Nashville* (Vol. 09, pp. 09-40-09-51). Metal Powder Industries Federation, Princeton, New Jersey.

Total number of authors:

3

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LUND UNIVERSITY

PO Box 117
221 00 Lund
+46 46-222 00 00

Advances in Powder Metallurgy & Particulate Materials—2012

Proceedings of the
2012 International Conference on
Powder Metallurgy & Particulate Materials
sponsored by the
Metal Powder Industries Federation
June 10–13, Nashville, TN

Compiled by

Ian Donaldson, FAPMI
GKN Sinter Metals

Nicholas T. Mares
Asbury Graphite Mills, Inc.



Metal Powder Industries Federation
105 College Road East
Princeton, New Jersey 08540-6692

Tel: (609) 452-7700 Fax: (609) 987-8523
www.mpif.org

Ignition temperatures for Cu-Al and Ni-Al reactions in elemental powder mixtures using Differential Scanning Calorimetry

Hossein Sina, Srinivasan Iyengar* and Solveig Melin
Materials Engineering
Lund University, Box 118
SE-22100 Lund
Sweden

*corresponding author (srinivasan.iyengar@material.lth.se)

ABSTRACT

The formation of intermetallic compounds in Cu-Al and Ni-Al systems has been studied in this work. Metal powders were mixed in stoichiometric amounts corresponding to CuAl_2 and NiAl . Pre-compacted as well as uncompacted metal powder mixtures were studied using simultaneous thermal analysis and thermogravimetry, with a protective atmosphere of flowing argon gas. Initiation and ignition temperatures for the reactions have been determined below the melting point of aluminium, at heating rates of 7.5° and 15°C per minute. The effect of parameters like particle size and compaction pressure on ignition temperature is presented. X-ray diffraction data on samples from *ex situ* studies confirm the presence of intermetallic compounds.

INTRODUCTION

It is well known that intermetallic compounds like various metal aluminides are materials suitable for high temperature and high performance applications¹. For example, nickel-aluminium compounds possess unique characteristics such as high melting point, low density, good load-bearing capacity and thermal conductivity as well as high oxidation resistance. The attractive combination of properties find their use in high temperature structural and coating applications in gas turbine components, thermal barrier coatings and surface catalysts²⁻⁴. Another example is that of copper aluminides which possess a high corrosion resistance combined with high stiffness. They are stable under different environments such as sea, air, steam, or a solution of dilute acids⁵. They can be used to produce gears, guide bushings, bearings, and structural components which are used in fresh and sea water as well as steam atmospheres⁵. The properties of aluminides are strongly dependent on the processing technique and parameters.

There are several routes for producing aluminide compounds such as conventional casting, rapid solidification, mechanical alloying, and powder metallurgical methods^{1,6}. Processes based on Self-propagating High temperature Synthesis (SHS) have attracted a lot of attention in the recent past. The SHS process can be used to form ceramics, intermetallics and composites at temperatures much above their melting points³. SHS has several advantages such as energy and time saving and can be used for different reactions like solid-solid reactions and solid-liquid reactions. In this approach, the reaction occurs quickly in a few seconds or minutes instead of hours for the conventional processes. The product from SHS also has high purity, as volatile contaminants are removed due to the high temperature produced during the reaction. However, the product is likely to have a relatively high porosity which may not be desirable in many applications. Higher pressures can be used during the reaction to decrease the amount of porosity^{2,6,7}. The SHS technique involves the ignition of a powder mixture in air or an inert atmosphere, producing an exothermic reaction that can be self sustaining⁸.

The maximum temperature achieved during the combustion reaction could exceed the melting temperatures for the components. There are two basic modes of SHS - the propagating mode in which the sample is ignited locally from one side. An external heating source (like a laser) can be used to initiate a local reaction which releases enough energy to enable the reaction front to propagate through the sample like a wave. In the thermal explosion mode, the whole sample is heated at a constant rate to a particular temperature (ignition temperature) at which the reaction is initiated and continues as a self-sustaining reaction¹. Thermal explosion is a good method to synthesize intermetallic compounds since it needs relatively lower processing temperatures, while the combustion mode is often used for the synthesis of refractory materials which require higher temperatures. Some studies have shown that the products synthesized from both methods have the same properties^{7,9}. Aluminides can be produced either by the propagating or thermal explosion method. A number of parameters can influence the SHS process, for example, the powder particle size and shape, heating rate, green density, and reaction atmosphere^{10,11}. These parameters can affect the reaction by altering the solid state diffusion mechanism¹¹ which is important in the pre-combustion stage. There are several studies focusing on the effect of the process parameters in different SHS synthesis methods for aluminides. It has been shown that by increasing the particle size, the reaction may not go to completion and the microstructure will consist of a mixture of different intermetallic compounds³. Small particle sizes with higher surface area, lead to better reaction kinetics and minimize diffusional effects. A high level of pre-compaction provides a large number of contacts between reactant particles, and this results in a stronger reaction. Therefore, thermal explosion is more likely to be initiated earlier than in samples with lesser density. It has also been reported that the ignition temperature decreases as the heating rate is increased¹.

Ozdemir et al² studied the production of NiAl by pressure assisted combustion synthesis. In this approach, the samples were first synthesized and consolidated simultaneously, followed by a heat treatment. It was observed that synthesis prior to the heat treatment resulted in the formation of different intermetallic compounds such as NiAl₃, Ni₂Al₃, and NiAl. Heating this sample to 1050°C for about an hour led to the formation of a homogeneous NiAl intermetallic compound with 99% density. Hunt et al¹² studied the effect of aluminium particle size on the ignition behavior of Ni-Al mixtures. A significant decrease in ignition time and temperature were observed in studies with nanoscale reactants. Plazanet et al⁶ investigated the effect of green density and heating rate on the combustion synthesis and microstructure of NiAl produced through SHS. They identified the various steps associated with the thermal explosion mode and observed that a higher heating rate can decrease the ignition temperature. The formation of NiAl intermetallic compounds by ball milling has been studied by Moshksar et al¹⁰. They observed that ball milling can induce an exothermic reaction between air and the activated reactant particles which could result in the early explosive formation of NiAl. Studies on the structure and properties of NiAl formed by SHS was carried out by Zhu et al⁴ which showed that single phase NiAl could be synthesized by induction heating. Kovalev et al¹³ studied transformations in a Cu-Al mixture corresponding to the stoichiometry Cu₃Al studied using a dynamic X-ray phase analysis method.

The binary phase diagrams for Cu-Al and Ni-Al systems are presented in Figs. 1&2. The Al-Cu system is characterized by a eutectic transformation occurring around 548°C, involving a liquid phase, an aluminium-rich solid solution (α) and the intermetallic compound CuAl₂ (θ -phase). It has been reported that thermal combustion occurs around the eutectic temperature at which the melt formation can stimulate the combustion reaction by wetting the surfaces of the reactants¹⁴. The product of this reaction is usually the θ phase (CuAl₂).

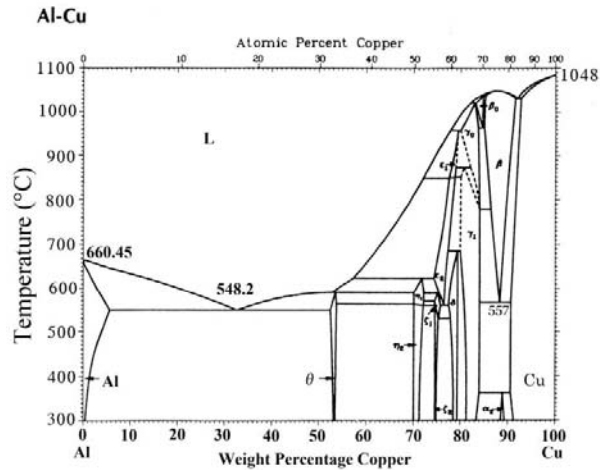


Figure 1. Al-Cu Phase diagram¹⁵

Fig. 2 shows the binary phase diagram for the Al-Ni system. The lowest temperature at which a melt can form in this system corresponds to the eutectic reaction which takes place at about 640°C and involves a liquid phase and an aluminium-rich solid phase as well as the solid intermetallic compound Al₃Ni. This temperature plays a critical role in the combustion synthesis of Al-Ni compounds. For Al-Ni mixtures, the combustion reaction initiates with the formation of a transient liquid phase which is typically a eutectic melt, and it spreads throughout the microstructure. Following, the wetting of the reactant particles by the melt, the combustion reaction is initiated leading to final product formation. Among all the phases in the Al-Ni system, the AlNi phase has the highest melting point and has good potential for high temperature applications. However, it has been shown that during the pre-combustion stage prior to liquid formation, low heating rates can lead to the formation of other compounds³.

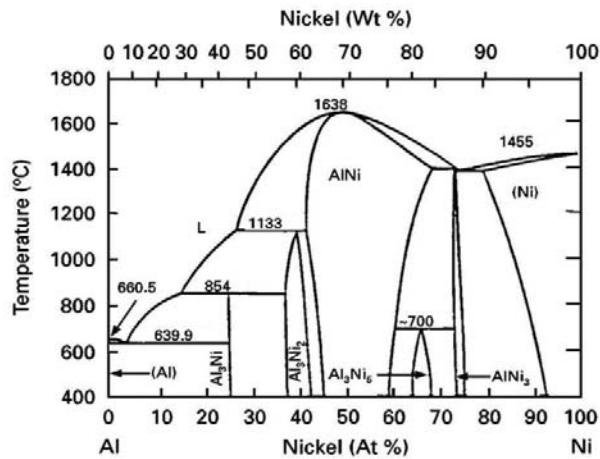


Figure 2. Al-Ni Phase diagram¹⁵

In this work, in order to get a deeper understanding of aluminide formation by SHS, ignition of Copper-Aluminium and Nickel-Aluminium powder mixtures has been studied using a Differential Scanning Calorimeter (DSC). The effect of particle size, heating rate, and compaction on the ignition behaviour of the samples has also been investigated.

EXPERIMENTAL

Samples have been made from elemental powders (pure aluminium, copper, and nickel) with different particle sizes (Table 1). Powders, in small quantities, were weighed and mixed in stoichiometric proportions corresponding to NiAl and CuAl₂.

Studies were made using powder mixtures, with and without compaction. Compacted disks were prepared using uniaxial compression in a 5 mm diameter steel die. The green density was approximately 70%.

Table 1. Maximum Particle size of elemental powders in the samples

Sample	Cu particle size (μm)	Ni particle size (μm)	Al particle size (μm)
Cu 5-Al15	5	-	15
Cu63-Al15	63	-	15
Cu 5-Al60	5	-	60
Cu63-Al60	63	-	60
Ni10-Al15	-	10	15
Ni45-Al15	-	45	15
Ni10-Al60	-	10	60
Ni45-Al60	-	45	60

A Simultaneous Thermogravimetry - Differential Scanning Calorimetry (TG-DSC) equipment (Netzsch, Model STA 449-F3 Jupiter) has been used for these studies. The DSC unit was calibrated for temperature and heat sensitivity using standard reference materials (pure metals). Baseline correction was done using data from heating cycles with empty crucibles. Compacted disks were placed in an alumina crucible on the sample holder. Loose powder mixtures were placed inside the crucible and the surface was made flat in each case. After closing the DSC chamber, it was evacuated and flushed with a reducing gas (Ar-4\%H_2) three to four times to minimize the presence of air and avoid oxidation of the sample during heating. Prior to introducing the purging/flushing gas into the chamber, it was passed through metal tubes packed with drying agents like anhydrous calcium chloride and molecular sieves to remove any moisture in the gas. The samples were heated at two different heating rates (7.5° and 15°C per minute) up to 645°C . After a holding time of 5 minutes at this temperature, the samples were cooled down to room temperature. The gas flow was maintained constant at 70 ml per minute during the whole experiment. Finally, the results were obtained as TG and DSC plots which could be analyzed to detect reactions and transformations. To identify the phases produced during the heating of the samples, X-ray diffraction (XRD) analysis was performed using a vertical Stoe Stadi MP machine equipped with a Germanium Monochromator (Johan geometry), a Copper tube and a Linear PSD as the detector in the transition mode. The samples were crushed to prepare powder samples which could be used for XRD studies.

RESULTS AND DISCUSSION

The experimental results obtained for Copper-Aluminium as well as Nickel-Aluminium samples are presented in the following section.

Cu-Al Samples

All the copper-aluminium samples of a given form (loose powder or disks) showed similar behavior with changes in heating rates for a given particle size combination. Fig. 3 shows typical DSC plots obtained for a Cu5-Al60 sample.

Fig. 3(a) shows typical data for disks, corresponding to a heating rate of 7.5°C per minute. It is seen that an exothermic peak appears first followed by an endotherm which is in turn followed by exothermic and endothermic peaks. The first exothermic peak can be attributed to the pre-combustion stage associated with solid-state interdiffusion of copper and aluminium particles and is accompanied by heat liberation. This could result in the formation of some solid solution phases as well as intermetallic compounds^{3,16}. The second peak, which is endothermic, occurs close to the eutectic temperature and is indicative of melting. The melt formed at this stage wets the particle surfaces and promotes combustion resulting in the third peak which is exothermic. The onset temperature corresponding to the third peak (exothermic) can be considered as the ignition temperature for the formation of the intermetallic compound CuAl_2 (θ -phase).

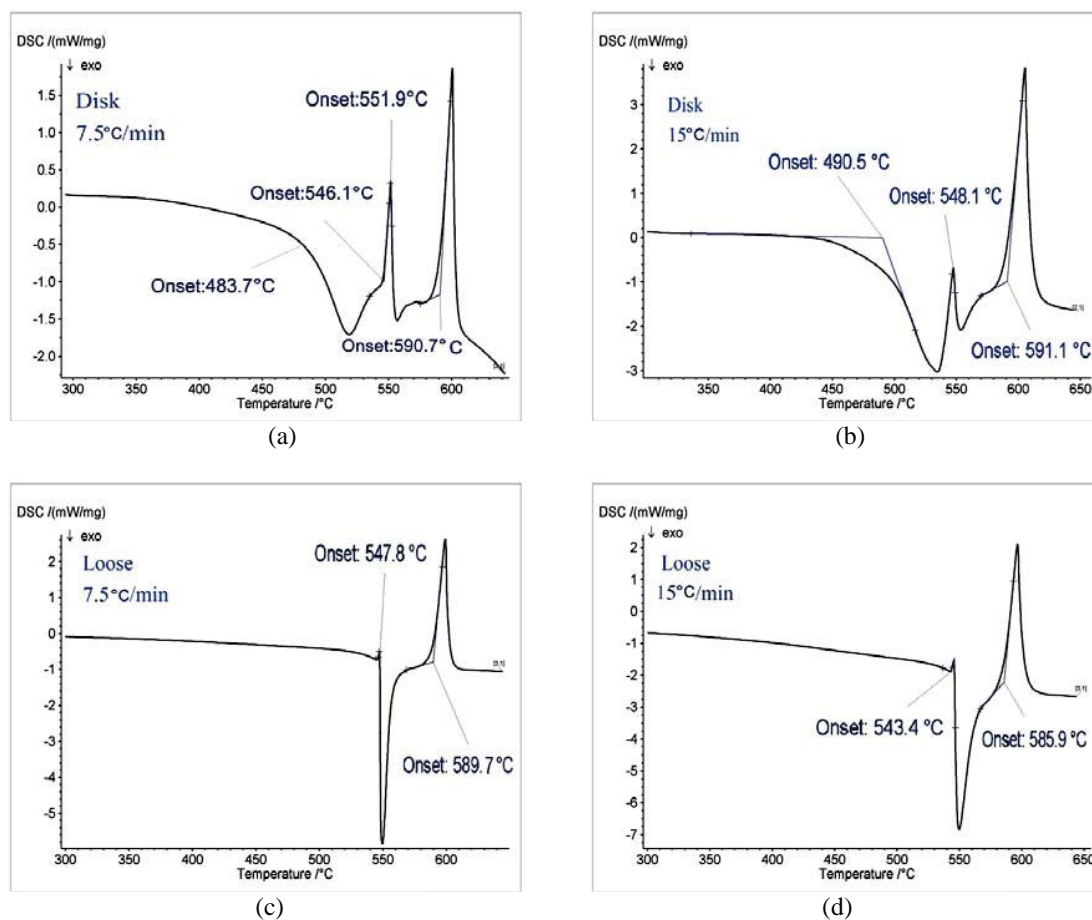


Figure 3. DSC data for Cu5-Al60 Samples: (a,b) Disk, 7.5° and 15°C/min; (c,d) Loose Powder, 7.5° and 15°C/min

According to the Cu-Al phase diagram, θ -phase melts at 591°C and an endothermic peak appears around this temperature (Fig. 3b,d).

Fig. 3 (b) shows data corresponding to a heating rate of 15°C per minute. After the first exotherm (pre-combustion), there is no endotherm corresponding to eutectic melting and subsequently the second exotherm corresponding to the ignition reaction appears, followed by the endotherm corresponding to θ -phase melting.

Fig. 3 (c&d) show the behavior of uncompacted Cu-Al samples at heating rates of 7.5° and 15°C per minute. These plots have only two peaks - an exothermic and an endothermic one. The exothermic peak corresponds to the main combustion reaction, which is occurring without any pre-combustion stage. Eutectic melting is also not observed and the only endothermic peak corresponds to the melting of the θ -phase.

The Pre-combustion stage:

Experimental observations clearly indicate the presence of a pre-combustion stage in compacted disk samples. However, this stage was absent in loose powder samples, presumably due to poor contact between the copper and aluminium particles which minimizes diffusion. Table 2 presents data on onset temperatures for pre-combustion in the copper-aluminium disk samples studied at two different heating rates. For a given sample, irrespective of the particle size combination, the onset temperature increases with increase in heating rate. This is understandable as the pre-combustion stage involves the interdiffusion of copper and aluminium in each other. A higher heating rate decreases the time for diffusion and leads to higher onset temperatures. It may be noted that, at 500°C, the coefficients for aluminium diffusion in copper and that of copper in aluminium are about 3.38×10^{-16} and $2.01 \times 10^{-17} \text{ m}^2 \text{ s}^{-1}$ respectively¹⁶.

Table 2. Onset temperatures for the pre-combustion stage in Cu-Al compacted disk samples

Sample	Onset temperature (°C)	
	7.5°C/min	15°C/min
Cu5-Al15	493.2	501.0
Cu5-Al60	483.7	490.5
Cu63-Al15	456.8	472.8
Cu63-Al60	473.3	475.6

Comparing the data for different particle size combinations at a constant heating rate, it is seen that the onset temperature is lower for the sample Cu5-Al60 relative to Cu5-Al15. The smaller copper particles (5µm) can encircle the larger aluminium particles (60µm) giving a good surface coverage and better Cu-Al contact, leading to better diffusion and lower onset temperatures. This effect is larger for the case where copper particles are large (53µm) relative to aluminium (15µm), due to contributions from the better interfacial contact and the faster diffusion of aluminium in copper. Keeping the aluminium particle size large, an increase in the particle size of copper also indicates a reduction in the onset temperature. This is perhaps due to the possibility of larger deformation of these particles during compaction, increasing the contact between copper and aluminium. For the case where the copper particle size is large, an increase in the aluminium particle size results in a higher onset temperature, perhaps due to the reduced contact area and mobility of aluminium.

From the DSC plots, the heats of various reactions can be calculated by computing the areas under the exothermic and endothermic peaks. The heat effects are referred to unit mass of the sample. However, in cases where partial conversions or multiple products are formed, it is not possible to determine the heat effects for a specific reaction or product. Hence, the enthalpy values are not reported here and the focus is on onset temperatures. However, it may be noted that Zaitsev et al¹⁷ reported a value of -15.82 kJ/mol (-176 J/g) as the enthalpy of formation for CuAl₂ at room temperature.

Eutectic melting:

As mentioned earlier, in all the compacted disk samples heated at 7.5°C per minute, the DSC curve showed an endothermic peak at 546.3±0.5°C. This is very close to the eutectic melting temperature 548°C in the Al-Cu binary phase diagram. Experimental data related to eutectic melting are shown in Table 3.

This peak indicates liquid formation due to eutectic melting. It was observed that compacted samples heated at the higher heating rate (15°C per minute), do not exhibit such a peak. As the heating rate is high, there is not enough time for the eutectic transformation to occur. Similarly, for uncompact samples, eutectic melting has not been observed. It may be noted that only the Cu5-Al60 uncompact sample showed a very small endothermic peak at 546° & 543.4°C, respectively at heating rates of 7.5° and 15°C per minute. This may be due to a better coverage of the large aluminium particle surface by the fine copper particles and the possibility of diffusion and absorption of copper in aluminium.

Table 3. Onset temperatures for the eutectic melting in Cu-Al compacted disk samples

Sample	Onset temperature (°C)	
	7.5°C/min	15°C/min
Cu5-Al15	545.8	No peak
Cu5-Al60	546.4	
Cu63-Al15	546.3	
Cu63-Al60	546.8	

Combustion phase:

Table 4 shows the onset temperatures for the combustion phase as a function of particle size and heating rate. These temperatures, considered as the ignition temperatures for the samples, increase when the reactant particles are relatively bigger. Samples containing small particles have a larger

surface area and tend to undergo the combustion reaction at lower temperatures. However, this effect seems to be more significant for uncompact samples in which the contact area between the copper and aluminium particles is relatively small and diffusional effects are small.

The combustion reaction needs enough heat to get initiated. Increasing the heating rate means that the sample will get a larger amount of heat in a shorter time, so this can initiate the ignition earlier. It is usually observed that increasing the heating rate leads to a lower ignition temperature.

Comparing the data for compacted disks to uncompact, loose powder samples, an increase in the ignition temperature is observed. This is due to the decrease in contact area between the particles. The deviation from this pattern observed for the Cu5-Al15 sample may be due to inhomogeneities in the mixture.

Table 4. Onset temperatures for the combustion stage in Cu-Al samples

Sample	Compacted		Loose powder	
	7.5°C/min	15°C/min	7.5°C/min	15°C/min
Cu5-Al15	548.9	541.8	549.5	543.9
Cu5-Al60	551.9	548.1	547.8	543.4
Cu63-Al15	553.6	544.5	571.1	568.9
Cu63-Al60	556.2	544.3	579.2	578.1

θ-phase melting:

According to the Cu-Al phase diagram, the intermetallic compound CuAl₂ (θ-phase) melts at 591°C. During the heating of the samples, an endothermic peak is therefore observed after the combustion stage. Table 5 presents such data for the Cu-Al samples. These data are quite close to the equilibrium value, for all particle sizes and heating rates.

Table 5. Onset temperatures for *θ-phase melting* in Cu-Al samples

Sample	Compacted		Loose powder	
	7.5°C/min	15°C/min	7.5°C/min	15°C/min
Cu5-Al15	589.9	589.0	589.3	586.1
Cu5-Al60	590.7	591.1	589.7	585.9
Cu63-Al15	591.0	588.6	588.4	592.6
Cu63-Al60	591.8	576.4	593.3	590.8

X-ray Analysis:

In order to confirm the phases formed during heating, a few samples were analyzed using X-ray diffraction. Fig. 4 presents the X-ray diffractogram obtained for the compacted disk (Cu5-Al60) sample which was heated in the DSC at 15°C per minute. The pattern indicates the presence of a single intermetallic compound (θ-phase) in the heated sample, indicating complete combustion.

In order to study the phases formed during the pre-combustion stage, another experiment was carried out on the same sample which was heated to 500°C at the rate 15°C per minute and it was held at this temperature for 1 hour and then cooled down. The X-ray diffractogram presented in Fig. 5 confirms the formation of θ-phase during the pre-combustion stage. However, aluminium was also identified in the pattern. Since this sample contained large aluminium particles, it is possible that the reaction has occurred mostly at the surface of these particles, while the core remained unreacted. On the contrary, copper particles are very fine, so a large number of them have participated in the reaction and have been consumed and therefore reflections corresponding to copper are not observed in the diffractogram.

For comparison purposes, a Cu5-Al60 sample in the uncompact condition was heated at the rate of 15°C per minute to 645°C, cooled and examined by X-ray diffraction. Fig. 6 indicates that, in addition to θ-phase, aluminium is present in the product, which may be due to the larger aluminium particles having an unreacted core.

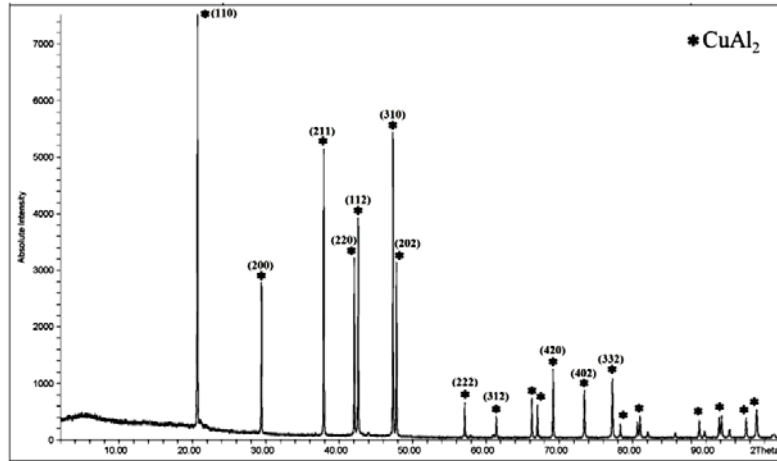


Figure 4. Diffractogram of the Cu5-Al60 compacted disk sample (15°C/minute).

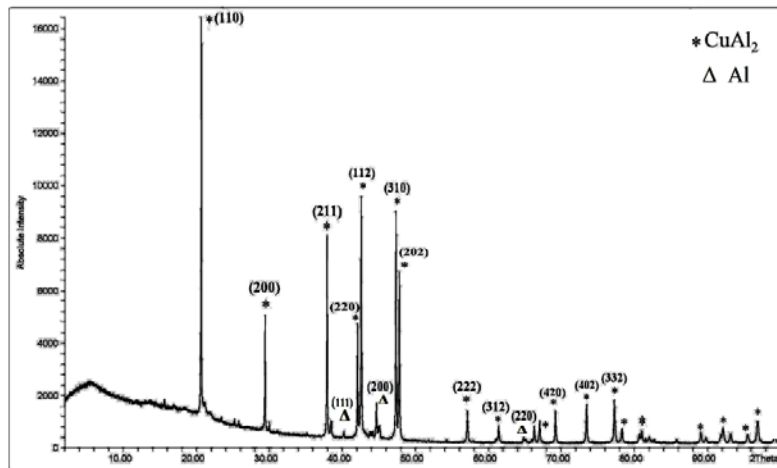


Figure 5. Diffractogram of the Cu5-Al60 compacted disk heated to 500°C (15°C per minute, holding time: 1 hour).

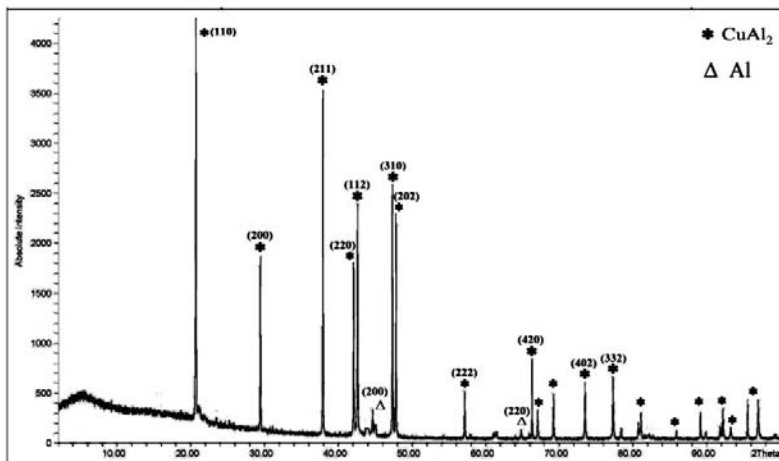


Figure 6. Diffractogram of the Cu5-Al60 uncompact sample (15°C/minute).

Ni-Al Samples

Typical DSC plots obtained for Ni10-Al60 samples in compacted as well as uncompact forms are shown in Fig. 7. The effect of heating rate on a disk or uncompact sample with a given particle size combination was found to be similar. From Fig. 7(a,b), two exothermic peaks are seen for the compacted samples heated at 7.5° and 15°C/min, while the uncompact samples display a single exothermic peak, Fig. 7 (c,d). The first exothermic peak corresponds to the pre-combustion stage and seems to be overlapping another peak, whereas the second one is very sharp and corresponds to the ignition reaction in the samples.

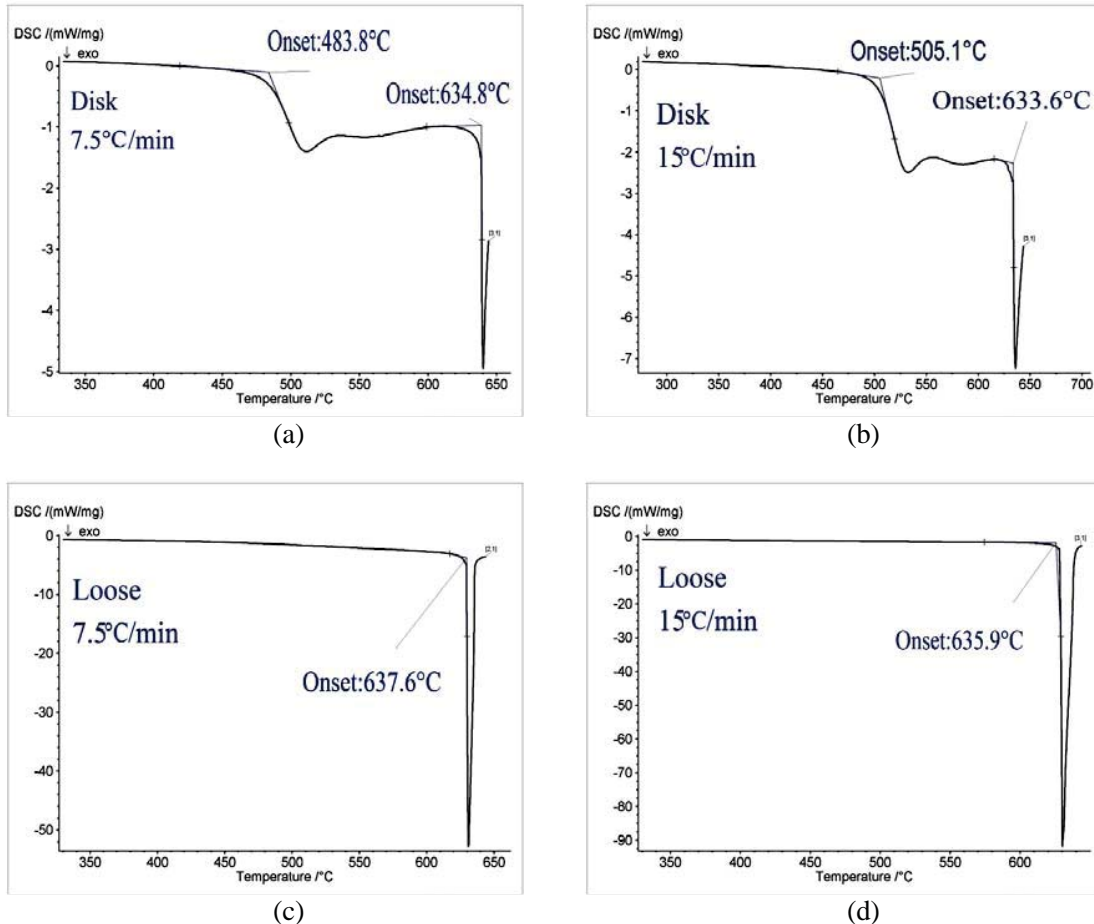


Figure 7. DSC data for Ni10-Al60 Samples: (a,b) Disk, 7.5° and 15°C/min; (c,d) Loose Powder, 7.5° and 15°C/min.

Tables 6 and 7 show the onset temperatures for these two exotherms. Uncompact samples did not show any pre-combustion peak, except for Ni45-Al60 which showed a moderate change in slope at 631°C. This is due to insufficient contact between the reactant particles which suppresses the pre-combustion reaction.

Table 6. Onset temperatures for the pre-combustion Stage in Ni-Al compacted disk samples

Sample	Onset temperature (°C)	
	7.5°C/min	15°C/min
Ni10-Al15	484.3	504.4
Ni10-Al60	483.8	505.1
Ni45-Al15	547.1	559.8
Ni45-Al60	571.2	589.9

From Table 6, it is seen that the onset temperature for the pre-combustion reaction is strongly influenced by the particle size of the two components, in particular nickel. Samples containing fine nickel particles have approximately the same onset temperature, while samples with larger nickel particles have significantly higher onset temperatures. Increasing the particle size of aluminum and nickel together, also led to an increase in the onset temperature. In order to have a low pre-combustion temperature, it is necessary to have small reactant particles. A higher heating rate initiates pre-combustion at higher temperatures. As noted before, a higher heating rate implies shorter diffusion times and hence the difficulty of initiation at low temperatures.

Table 7 presents data on the second exothermic peak in the DSC plots. This corresponds to the main combustion reaction resulting in intermetallic compound formation. For all the samples studied, the onset (ignition) temperatures for combustion lie very close to the eutectic melting temperature of 639.9°C in the aluminium rich end of the Al-Ni phase diagram. The solid phases involved in this transformation are the α -solid solution and the intermetallic compound Al_3Ni . Although liquid formation and stimulation of combustion are possible, no endothermic peak was observed near the onset temperatures. However, masking of the small endothermic effect by the large exothermic heat due to combustion cannot be ruled out. Increasing the heating rate decreases the ignition temperature and is comparable to the behavior discussed in the case of Cu-Al mixtures. Samples containing small particles or a combination of large and small reactant particles, exhibited lower ignition temperatures. Most of the compacted disks, have lower ignition temperature as compared to uncompacted samples. However, this difference is not significant in some cases. The exothermic heat of combustion observed experimentally depends on the intermetallic compounds formed and their heats of formation. For NiAl, Nash et al¹⁸ report value of -61.8 ± 1.1 kJ/mol (-721 J/g) at room temperature. However, previous studies have shown the formation of several different intermetallic compounds during the heating of Ni-Al mixtures¹⁻³. Because of these uncertainties, this study focuses on the onset temperatures.

Table 7. Onset temperatures for the combustion stage in Ni-Al samples

Sample	Compacted		Loose powder	
	7.5°C/min	15°C/min	7.5°C/min	15°C/min
Ni10-Al15	636.9	634.1	638.1	635.2
Ni10-Al60	634.8	633.6	637.6	635.9
Ni45-Al15	635.8	635.1	635.3	634.7
Ni45-Al60	636.2	636.4	638.2	637.4

Fig. 8 shows the diffractogram for the Ni10-Al60 compacted disk sample heated at the rate of 15°C per minute. The pattern confirms the presence of several intermetallic compounds such as Al_3Ni , Al_3Ni_2 , and NiAl in the final product. In addition, a few other compounds like Al_2O_3 and AlH_3 have also been detected. This is due to the interaction of the sample with the gas mixture and needs to be suppressed completely.

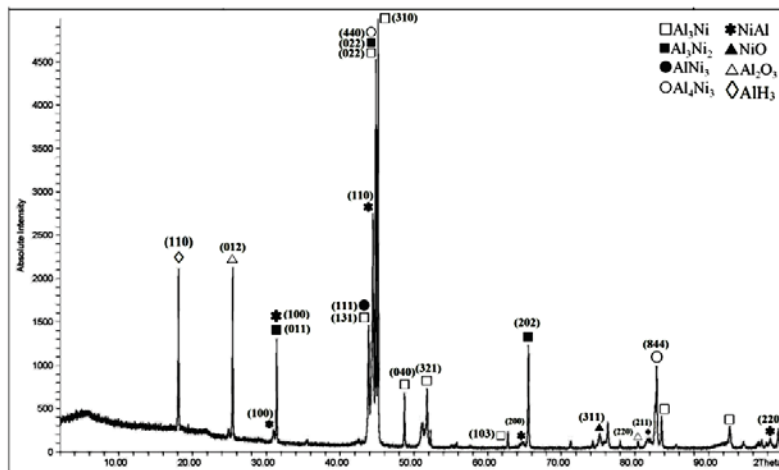


Figure 8. Diffractogram of the Ni10-Al60 compacted disk sample (15°C/min)

CONCLUSION

In this paper, the combustion synthesis of nickel and copper aluminides has been studied using a differential scanning calorimeter. The effect of different parameters such as heating rate, pre-compaction, and particle size on the ignition behavior of samples has been investigated. For compacted samples, the results confirm the presence of a pre-combustion stage involving the formation of solid solutions and intermetallic compounds through solid-state diffusion. This is followed by eutectic melting and combustion leading to the formation of intermetallic compounds. For some uncompact samples, the pre-combustion stage and eutectic melting were not observed. Increasing the heating rate lowered the ignition temperature of the samples. Small particles and a combination of small and large particles lead to lower reaction temperatures. X-ray diffraction studies confirm the presence of a single intermetallic compound (CuAl_2) after the pre-combustion and combustion stages in a Cu-Al mixture. Multiple intermetallic compounds have been observed to form during the heating of a Ni-Al mixture.

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