Effect of cooling rate on solidification of Al-Ni alloys

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Abstract. Particles of Al-Ni alloys with different compositions (Al–50 wt-% Ni and Al–36 wt-
% Ni) were produced using a drop tube-impulse system, known as Impulse Atomization. The
microstructure of these rapidly solidified particles was compared with those solidified in a
DSC at low cooling rates (0.083 and 0.33 K/sec). Also, the microstructure of the sample
solidified in microgravity on-board of the TEXUS 44 sounding rocket was analyzed. Neutron
diffraction was used to investigate the phases formed during different solidification processes.
From SEM micrographs and neutron diffraction it was found that the inner parts of the TEXUS
sample and the sample that was cooled at 0.083 K/sec contain almost no eutectic structure. The
outer rim of the TEXUS sample showed the highest amount of Al3Ni and lowest amount of
Al3Ni2. Increasing the cooling rate from 0.083 to 0.33 K/sec increased the Al3Ni/Al3Ni2 ratio.
Opposite trend was observed in the impulse-atomized particles, where increasing the cooling
rate decreased the Al3Ni/Al3Ni2 ratio.

1. Introduction
The materials characteristics are greatly influenced by microstructure evolution during the
solidification, which is a function of processing parameters during production. A common
solidification reaction in industrial alloys, such as Fe-C steels and Al-, Cu- and Ti-based alloys is the
peritectic reaction [1]. Aluminum alloys with nickel are practically significant due to their high
temperature strength and heat resistance [2] and also for their catalytic capability [3]. However, it has
been shown that the final properties of the alloy depend on its constituent phases formed during the
solidification process. For example, the activity of Raney nickel catalysts is strongly affected by the
amount and morphology of the Al3Ni [3-4].

Recently, modeling of the solidification of Al-rich Al-Ni alloys has attracted the attention of many
scientists who try to understand and simulate this important and complex system [5-8]. On the other
hand, many different experimental results have been published using different techniques to
understand the effect of various processing parameters on the final phase fractions and the evolved
microstructure [9-11]. Bao et al. [9] analyzed Al–Ni powders produced by gas atomization having
different compositions on the Al-rich side of the phase diagram. They used neutron diffraction (ND)
and X-ray diffraction (XRD) to identify the existing phases at the surface and in the bulk of the
particles. Their results show a significant dependence of phase selection during the solidification of
different sized gas atomized particles. Using impulse atomization technique, it has been shown that in
Al-50 wt-%Ni, by increasing cooling rate, the ratio of Al$_3$Ni to Al$_3$Ni$_2$ decreases, while opposite behavior was observed in Al-36 wt-%Ni [10-11].

Although atomization techniques offer containerless undercooling in conjunction with high degrees of cooling rates, the measurement of the whole history of nucleation and growth in these techniques is difficult. Therefore, electromagnetic levitation has been extensively used to containerlessly undercool bulk samples to study the solidification of a freely suspended droplet but at small cooling rates. The unique possibility of undercooling bulk samples due to the avoidance of heterogeneous nucleation on container walls coupled with accessibility of the solidifying droplet for direct observation and temperature measurement make the electromagnetic levitation technique a powerful tool for solidification studies [12]. This technique was used to study the effect of melt undercooling prior to solidification on the dendrite growth velocity of various Al-Ni alloys. The results showed that while on the Ni-rich side, growth velocity increases with increasing undercooling, Al-rich alloys show an unusual decrease in growth velocity with increasing undercooling [13]. Since it was already known that the melt stirring due to electromagnetic forces can generate pronounced forced convection [14], which in turn would affect the solidification, it was decided to perform the experiment in microgravity. Microgravity conditions provide the advantage of reduced electromagnetic stirring which is essential for the investigation of nucleating kinetics and growth in the absence of convection. The experiment was performed during the sounding rocket mission TEXUS 44 of the European Space Agency (ESA) and the German Aerospace Center (DLR). TEXUS sounding rocket missions provide an excellent quality of reduced gravity of the order of 10^{-4}g [15]. An Electro-Magnetic Levitator (EML) for use in reduced gravity environment (TEMPUS-EML) was utilized to process the sample in reduced gravity [16].

In this paper, solidification of Al-50 wt-%Ni and Al-36 wt-%Ni produced using different techniques will be studied. Solidification of both alloys in slow cooling rate condition in a furnace, high cooling rate condition during free-fall in a drop tube and high undercooling condition in reduced gravity on-board of the TEXUS 44 rocket will be discussed.

2. Experimental

2.1. Impulse atomization

Two different compositions of Al-Ni alloys (36 and 50 wt-% Ni) were produced by melting 99.9% pure Al and Ni (Alfa Aesar) in an induction furnace located at the top of 4 meters high drop tube. Each alloy was held for 30 minutes at 100 K above its liquidus temperature. Then, the liquid was pushed through small nozzles placed at the bottom of the crucible. In this technique, known as Impulse Atomization (IA), a liquid jet is generated when the molten metal emerges from the nozzles. Then, it breaks up into spherical droplets due to Rayleigh instability. The falling droplets lose heat to the surrounding stagnant gas (helium) and solidify before reaching the bottom of the atomization vessel. The details of this technique can be found elsewhere [17]. The solidified particles were washed and sieved into different size ranges, from 100 to 1000 μm, according to Metal Powder Industries Federation (MPIF) standard 05 [18].

Neutron diffraction (ND) was used to characterize the phases formed during solidification. The experiments were conducted using a neutron beam of 1.33 Å wavelength at National Research Council of Canada - Canadian Neutron Beam Centre (NRC-CNBC) in Chalk River, ON, Canada. To study the microstructure, Scanning electron microscopy was then performed using Zeiss Evo MA15 SEM with 20 keV electron beam energy.

2.2. Solidification at Low Cooling Rates

In order to compare the microstructure of IA samples to those solidified in near equilibrium conditions, 30 mg of IA particles of Al-36wt-%Ni and Al-50wt-%Ni with diameter of 750 μm were
completely melted again by increasing the temperature to 1773 K. This was done in a Setaram Labsys
Evo Differential Scanning Calorimetry (DSC). The DSC had been calibrated for temperature and heat
measurement for the entire temperature range using standard samples of Zn, Sn, Al, Ag, Au and Ni.
The molten samples solidified at two different cooling rates of 0.083 K/sec and 0.33 K/sec. The
microstructure of as solidified particles was studied using SEM.

Image analysis was performed on the SEM micrographs of the impulse-atomized particles and
those solidified using DSC to find out the volume fraction of each phase. This analysis was performed
by ImageJ, a public domain Java image processing program [19].

2.3. Solidification under Microgravity
The TEMPUS facility, which is designed for electromagnetic levitation (EML) in reduced gravity
[16], has been integrated into a sounding rocket. TEXUS sounding rockets provide about 320 seconds
of reduced gravity time. The crystallization front velocity of Al-50wt-%Ni in the undercooled liquid
phase was measured during the TEXUS44 sounding rocket flight [9]. Three undercooling and
solidification cycles were obtained. The sample was heated by more than 200 K above the liquidus
temperature of this alloy in order to reduce and even eliminate Al-Oxides at the surface of the liquid
drop, which can act as heterogeneous nucleation sites of high catalytic potency. The sample
undercooled during the subsequent solidification cycles to $\Delta T_1 = 185$ K and $\Delta T_2 = 220$ K, respectively.
The TEXUS 44 sample was then studied using SEM and neutron diffraction. Image analysis using the
software ImageJ was also used to find the volume fraction of the phases in the TEXUS sample.

3. Results and Discussion

3.1. Solidification at slow cooling rates
Figure 1 and 2 show the microstructure of Al-50 wt-%Ni particles solidified at cooling rates of 0.083
K/sec and 0.33 K/sec, respectively. It can be observed that in these figures the microstructure is
slightly refined at the higher cooling rate.

In both figures, Al$_3$Ni$_2$, the light grey phase, and Al$_3$Ni, the dark grey phase, were identified using
EDX. No sign of the primary AlNi phase was observed in these samples. This will be further
discussed in 3.2. It was also noticed that in the particles solidified under the lower cooling rate (0.083
K/sec), visually no eutectic structure can be found. On the other hand, in the particle that solidified at
0.33 K/sec cooling rate, eutectic microstructure can be seen (Figure 2). It must be noted that the
equilibrium phase diagram does not predict any eutectic transformation for this alloy. This will be
discussed in the section “Phase quantification”. On the other hand, microstructural analysis of Al-36
wt-%Ni particles solidified with cooling rates of 0.083 K/sec and 0.33 K/sec also showed that the
particle cooled at higher cooling rate has finer microstructure. At both cooling rates, solidified
particles contain the primary phase Al$_3$Ni$_2$, the peritectic phase Al$_3$Ni and the eutectic. It is clear that
for this composition even at such low cooling rates the peritectic reaction of Liquid+Al$_3$Ni$_2$ $\rightarrow$ Al$_3$Ni
does not go to completion. This is not surprising since peritectic reactions need diffusion in solid and
therefore behave very sluggish [20].

3.2. Solidification at high cooling rates
It is known that impulse atomization technique produces rapidly solidified particles [17].
Microstructure of an impulse-atomized particle of Al-50 wt-%Ni with the diameter of 328 $\mu$m is
shown in Figure 3. Clearly, the microstructure is significantly finer than those in Figures 1 and 2. EDX
analysis of the phases observed in Figure 3 showed that the white phase, light grey phase and dark
grey phase are indeed Al$_3$Ni$_2$, Al$_3$Ni and eutectic structure, respectively and no sign of AlNi was
observed. Shuleshova et al. [21] by in situ X-ray measurements on highly undercooled levitated
droplets showed that in Al-50 wt%Ni even under non-equilibrium conditions the primary phase is
AlNi. It obviously decomposes on further cooling as it is not observed in the final microstructure. The
observed microstructure will be discussed in the section “Phase quantification”.

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**Figure 1.** SEM micrograph of Al-50 wt-%Ni particles solidified under cooling rates of 0.083 K/sec.

**Figure 2.** SEM micrograph of Al-50 wt-%Ni particles solidified under cooling rates of 0.33 K/sec.
3.3. Solidification in reduced gravity

The temperature-time profile of the TEXUS sample, Al-50 wt-%Ni alloy, is shown in Figure 4. The spikes on the temperature curve at the beginning of the experiment are due to oxides that were still on the sample’s surface. Since oxides show in general a higher emissivity than metals the pyrometer signal changes abruptly each time when an oxide layer on the surface of the rotating sample moves thru the observation window of the pyrometer. After oxides were eliminated by increasing the temperature, the oscillations of the temperature signal vanished and the sample was cooled until the first recalescence was completed. Then the sample was completely melted again for the second cooling cycle. During this cycle, and shortly after the first recalescence, the reduced gravity period was finished. The primary phase undercooling achieved was $\Delta T_1=185$ K and $\Delta T_2=220$ K for the first and second cycles, respectively. The cooling rate achieved by the sample before the first recalescence of the second cycle was inferred from the temperature-time profile as 14 K/sec, assuming that the slope of the cooling curve was constant in the last 200 K prior to the start of solidification. The cooling rate was further decreased after the first recalescence to be 10 K/sec.

The growth velocity of the solidification front in the undercooled liquid was also measured. The result of this measurement was presented elsewhere [13]. In brief, performing the experiment on Al-50 wt-%Ni on-board of the TEXUS 44 sounding rocket resulted in observation of two significant and important differences between the experiments in normal gravity and in reduced gravity. First, the growth velocity measured in reduced gravity is substantially smaller than the data taken under 1g conditions. Second, in reduced gravity, it was apparent that the growth velocity increases with increasing undercooling as usual for a great variety of alloys. In this section the solidification microstructure of the TEXUS 44 specimen will be discussed.

Microstructural analysis of the solidified specimen showed a distinct difference between the inner part and outer rim of the particle. The microstructure of the outer rim of the TEXUS sample is shown in Figure 5a and 5b. The outer rim of the sample contains $\text{Al}_3\text{Ni}_2$ primary dendrites, which are surrounded by the peritectic phase $\text{Al}_3\text{Ni}$ and an interdendritic Al-rich eutectic.
Figure 4. The temperature-time profile of the TEXUS 44 specimen. The arrows are:
1. Start of first cooling cycle, 2. First recalscence of the first cycle, 3. Start of second cooling cycle, 4. First recalscence of the second cycle, 5. End of microgravity, 6. End of temperature measurement period.

Figure 6 shows an SEM micrograph of the inner part of the Al-50 wt-%Ni sample processed during TEXUS 44 flight (in the following denoted as TEXUS sample). The inner part is characterized by a large amount of porosity, the absence of the Al-rich eutectic and dominant occurrence of the Al₃Ni₂ phase.

Neutron diffraction was used to investigate the phases formed within the impulse-atomized particles and the TEXUS sample. Figure 7 shows a small range of the diffractograms from all samples superimposed on each other. All peaks found in the diffractogram correspond to the phases Al, Al₃Ni and Al₃Ni₂.

From Figure 7 it is evident that the TEXUS sample is missing the characteristic (111) peak of Al solid solution phase, a component of the eutectic, at 2.68 Å⁻¹. Given the high depth of penetration of neutrons in the sample and the absence of Al phase peak in the diffraction pattern, clearly if any Al phase is present in the sample, it is below the ND detection limit, which is about 0.5 vol.% [22].

3.4. Phase quantification
SEM micrographs from the samples solidified in the DSC, impulse-atomized particles and the TEXUS samples were further investigated using ImageJ to find the volume fraction of consisting phases. For this purpose, from each of the samples solidified in the DSC, ten particles were used for image processing. The average of the phase fractions were then taken and reported as the volume fraction of each phase.

For impulse-atomized samples, twelve images from each particle size were used to measure the volume fraction of phases in each particle size. Then, the secondary dendrite arms spacing was measured and the cooling rate achieved for each particle size was estimated [10-11] using the coarsening model proposed by Kurz and Fisher [23].
Figure 5. a) SEM backscattered electron image of the TEXUS Al-50 wt-%Ni specimen. Outer rim and inner part. b) Outer rim of the specimen shown in Figure 5a in higher magnification.
Figure 6. SEM backscattered electron image of the inner part of the TEXUS solidified Al-50 wt-%Ni specimen exhibiting the Al$_3$Ni$_2$, light grey area, and Al$_3$Ni phase, dark grey area. The black areas represent voids.

Figure 7. Part of neutron diffractogram of various particle sizes of impulse-atomized Al-50 wt-%Ni and the TEXUS sample, showing the absence of the Al peak in the TEXUS sample.
The secondary dendrite arm spacing was related to the cooling rate using Eq. 1 and Eq. 2 for Al-50 wt\%Ni and Al-36 wt\%Ni, respectively [11].

\[
\lambda = 75.38 \dot{T}^{-0.33}
\]  
(3)

\[
\lambda = 69.58 \dot{T}^{-0.33}
\]  
(4)

The volume percent of the phases in the inner part and outer rim of the TEXUS sample were also measured from the SEM images of each part. For the cooling rate, from Figure 4, it was found that the maximum cooling rate was achieved at the beginning of the solidification, 14 K/sec, which then declined to 10 K/sec after the first recalcitrance. It is expected that this cooling rate goes further down as more solid forms and releases its latent heat; and also, as the temperature gradient between the droplet and the environment decreases.

Figure 8 shows the Al\textsubscript{3}Ni to Al\textsubscript{3}Ni\textsubscript{2} ratio that was found from image analysis and was plotted as a function of cooling rate. The data for TEXUS sample is drawn with a straight line for each of the inner parts and the outer rim. Both lines were cut off at 14 K/sec since that was the maximum cooling rate achieved. Also on this figure, the results reported by Patchett and Abbaschian [26] are shown by circles along with the technique that was used to produce the sample.

**Figure 8.** The Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} ratio as a function of cooling rate from the image analysis on samples of Al-50 wt-%Ni (filled symbols) and Al-36 wt-%Ni (empty symbols). Different symbols represent different production techniques: DSC (at two cooling rates of 0.08 K/sec, shown by diamonds, and 0.33 K/sec, shown by squares), impulse atomization, shown by triangles, and EML during TEXUS 44 campaign, shown by dashed lines, (only Al-50 wt-%Ni), as well as from the literature, shown by circles. The line with arrows represents the cooling rate achieved by the TEXUS sample before solidification.
The first striking feature of Figure 8 is the similarity of the Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} ratio in the sample that was cooled at 0.083 K/sec cooling rate in the DSC and that of the inner parts of the TEXUS sample. Also, as it was mentioned in the previous sections, in both samples the eutectic was not observed. Understanding the similarity of the Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} ratio in the inner parts of the TEXUS sample to that of the slow cooled sample requires better understanding of the solidification of both samples. Possible solidification scenarios are now discussed.

The sample that was cooled in the DSC, experienced prolonged time at high temperatures resulting in extended coarsening of the primary phase. This leaves a little surface area for the nucleation of the peritectic phase Al\textsubscript{3}Ni. On the other hand, due to extensive coarsening of the primary phase there is not enough liquid for peritectic reaction to proceed further. These hypotheses can explain why the ratio of Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} is much smaller than that predicted by the equilibrium phase diagram (~0.89). Increasing the cooling rate, by almost one order of magnitude in the DSC, results in less coarsening and finer structure, which leaves more surface area for Al\textsubscript{3}Ni nucleation. That can explain the increase in the Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} ratio. However, larger cooling rate does not allow for further continuation of Al\textsubscript{3}Ni growth, as the temperature reaches the eutectic temperature. Therefore, the remaining liquid solidifies into eutectic microstructure (Figure 2).

In the TEXUS sample, after it experiences 220 K primary phase undercooling, solidification starts from the surface, since the heat transfer is highest on the surface, and solid grows towards the inner parts. The initial growth rate is high because of high level of undercooling. However, this high growth rate decreases rapidly as the recalescence increases the sample temperature and decreases the cooling rate. Tourret et al. [5] showed that in Al-42 wt%Ni less than 10 vol.% of the solid formed by the end of the recalescence period in which the growth rate is the fastest, while more than 60 vol.% of the solid formed after the recalescence and before the peritectic reaction L+Al\textsubscript{3}Ni\textsubscript{2}→Al\textsubscript{3}Ni. The growth rate in this stage is much slower than that during the recalescence. It is expected that the amount of solid formed before the peritectic reaction to be higher in Al-50 wt%Ni alloy. It is argued that the decreasing cooling rate as a result of increasing solid formation and poor heat extraction from the large droplet (~6mm diameter) in the EML chamber [24-25], resulted in the formation of large amount of Al\textsubscript{3}Ni\textsubscript{2}. Therefore, similar to the sample solidified in the DSC, there are not enough surfaces available for the nucleation of Al\textsubscript{3}Ni. That is why the ratio of Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} in the inner parts of this sample is low. It is also expected that most of the remaining liquid in the small interdendritic regions to be consumed by the peritectic reaction, and thus, no eutectic region is seen in this sample.

From Figures 8 it is clear that the value of Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} ratio in the outer rim of the TEXUS sample is significantly higher than that of other samples studied in this research. As it was discussed, it is expected that the solidification starts from the surface at high growth rate. As a result, the microstructure in this region is refined and more surface area is available for the Al\textsubscript{3}Ni nucleation. However, this cannot justify the high volume fraction of Al\textsubscript{3}Ni in this region. It is known that the heat treatment of the Al-Ni samples results in an increase in Al\textsubscript{3}Ni fraction [26]. It is argued that since this region acted as the heat passage for the entire volume of the specimen, a phenomenon similar to heat treatment has occurred to the phases in the outer rim.

On the other hand, Figure 5 shows that the outer rim of the TEXUS sample contains eutectic structure. During solidification, slow growth of Al\textsubscript{3}Ni phase coupled with the higher cooling rate in the outer rim does not allow for further continuation of Al\textsubscript{3}Ni growth, which in turn can result in remaining of liquid to transform to eutectic structure.

Impulse atomization on the other hand can effectively extract the heat from the solidifying droplet. Extensive refinement of the microstructure as a result of high cooling rate and undercooling due to containerless processing provides massive surface area for the Al\textsubscript{3}Ni to nucleate. However further increasing the cooling rate results in reduction of Al\textsubscript{3}Ni/Al\textsubscript{3}Ni\textsubscript{2} ratio. This can be explained by the slow growth rate of Al\textsubscript{3}Ni phase. It should be noted that the effect of cooling rate on the phase fractions is two-fold; (1) it affects the nucleation and growth rate of each phase and (2) it refines the microstructure of the primary phase, which in turn increases the interface area between solid and liquid and influences the heterogenous nucleation of the peritectic phase [27]. These competing
phenomena can define the ratio of the phases within the droplets.

Comparing the results of the techniques used in this research with those reported by Patchett and Abbaschian [27] confirms the importance of the processing parameters in the phase fractions of a solidified specimen. While the $\text{Al}_3\text{Ni}/\text{Al}_3\text{Ni}_2$ ratio of the two samples that they studied in a containerless system such as EML and drop tube follows similar trend to that of the TEXUS and impulse-atomized samples, the results from the quenched and splat cooled samples are significantly different. This may be due to the constrained growth of the solid in the quenched and splat cooled samples, in which the released latent heat is extracted through the solid. In containerless techniques such as EML, impulse atomization or drop tube, however, the latent heat is extracted through the undercooled liquid. This can result in different levels of undercooling (primary phase, peritectic and eutectic) for the solidifying phases. Considering the complexity of the Al-Ni system with two peritectic and one non-equilibrium eutectic transformations it is not surprising that the different levels of undercooling achieved in different techniques resulted in different trend of changes in $\text{Al}_3\text{Ni}/\text{Al}_3\text{Ni}_2$ ratio. Also, the possibility of an anisotropic structure coupled with measurements using microscopy techniques may lead to errors and large scatter in measurements. Further work using 3D quantitative characterization techniques, such as neutron diffraction, on samples solidified using processes that result in constrained growth is needed.

In the case of Al-36 wt-% Ni, increase of the cooling rate from 0.083 K/sec to 0.33 K/sec did not have a significant effect on the $\text{Al}_3\text{Ni}/\text{Al}_3\text{Ni}_2$ ratio. Increasing the cooling rate to that of impulse-atomized droplets seems to initially decrease the ratio. However, further increasing the cooling rate resulted in higher ratio. In the lower cooling rate regime, such as those in the DSC, increasing the cooling rate refined both $\text{Al}_3\text{Ni}_2$ and $\text{Al}_3\text{Ni}$, which did not affect the ratio significantly. Further increasing the cooling rate may result in the reduction of the ratio as the growth of $\text{Al}_3\text{Ni}$ is sluggish. However at high cooling regime, such as in impulse-atomized droplets, increasing the cooling rate results in suppression of the primary $\text{Al}_3\text{Ni}_2$. It is also shown that the cooling rate required for suppression of primary $\text{Al}_3\text{Ni}_2$ decreases as the composition moves toward the peritectic liquidus temperature [27].

4. Summary

Two different compositions of Al-Ni alloys (36 and 50 wt-% Ni) were solidified under different conditions. Slow cooling rate in a DSC, impulse atomization in a drop tube. Also, Al-50 wt-% Ni was solidified on-board of TEXUS 44 sounding rocket using the TEMPUS EML facility for containerless processing of liquid metals in reduced gravity.

From the SEM micrographs it was found that both the inner parts of the TEXUS sample and the sample that was cooled at 0.083 K/sec in a DSC contain almost no eutectic. Neutron diffraction confirmed this observation.

The outer rim of the TEXUS sample showed the highest amount of $\text{Al}_3\text{Ni}$ and lowest amount of $\text{Al}_3\text{Ni}_2$. This observation was attributed to the reheating of $\text{Al}_3\text{Ni}_2$ in that zone during post-recalescence period.

Increasing the cooling rate from 0.083 K/sec to 0.33 K/sec in DSC experiments resulted in the formation of some eutectic microstructure and it increased the $\text{Al}_3\text{Ni}/\text{Al}_3\text{Ni}_2$ ratio. Opposite trend was observed in the impulse-atomized particles at higher cooling rates, where increasing the cooling rate decreased the $\text{Al}_3\text{Ni}/\text{Al}_3\text{Ni}_2$ ratio.

In Al-36 wt-% Ni, increasing the cooling rate from 0.083 K/sec to 0.33 K/sec did not significantly change the volume fraction of the respective phases. However in the impulse-atomized sample the $\text{Al}_3\text{Ni}/\text{Al}_3\text{Ni}_2$ ratio was increased.

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