Phase quantification of rapidly solidified Al-Ni powders using Rietveld analysis

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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 [1]. 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.

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. Profile refinement was carried out using the software GSAS on ND patterns. Three phases (i.e. Al₃Ni₂, Al₃Ni and Al) were identified. Figure 1 shows an example of measured ND pattern of 5 grams of IA particles with diameter of 500 µm along with the calculated diffraction pattern. The top line shows the calculated diffraction pattern as obtained from GSAS software and the bottom line represents the difference between the observed and calculated diffraction pattern. The few and low oscillation peaks in this curve indicate a satisfactory agreement between measured and refined patterns.

The Al_3Ni to Al_3Ni_2 ratio that was found from Rietveld refinement is shown in Figure 2.

Impulse atomization 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 Al₃Ni to nucleate. However further increasing the cooling rate results in reduction of Al₃Ni/Al₃Ni₂ ratio. This can be explained by the slow growth rate of Al₃Ni 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. These competing phenomena can define the ratio of the phases within the droplets.

References

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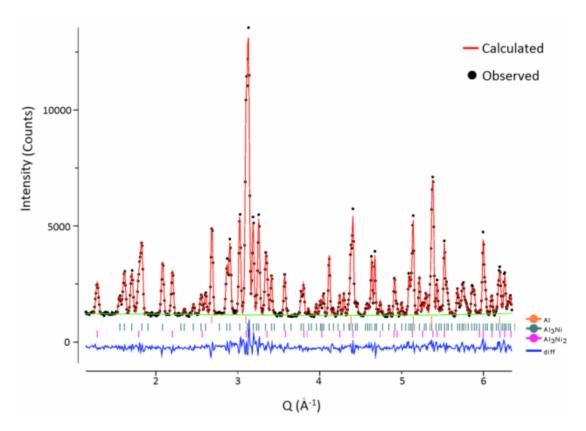


Fig. 1 Profile refinement of Al-36 wt%Ni with the diameter of 500 μm using the GSAS computer code.

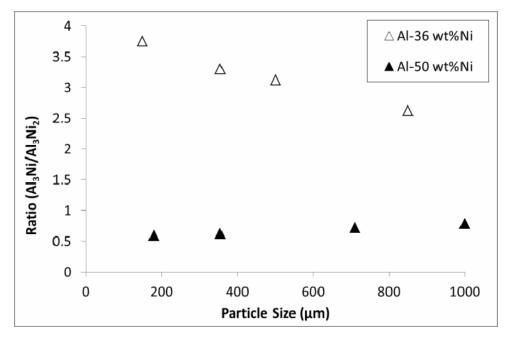


Fig. 2 The Al3Ni/Al3Ni2 ratio as a function of cooling rate for two Al-Ni alloys.