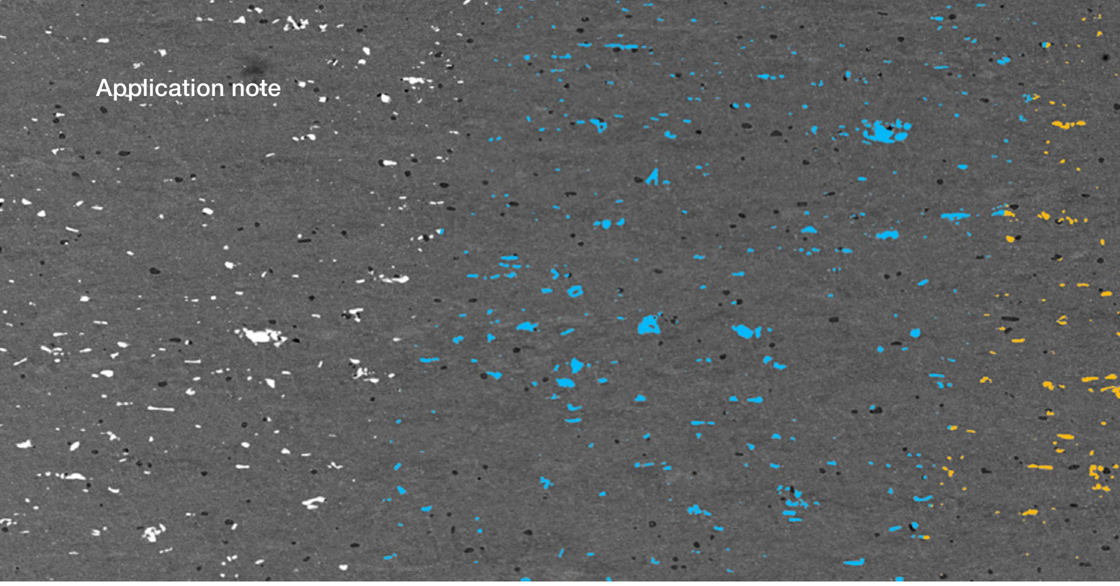
Application note



Automated SEM analysis of intermetallic particles in aluminum

Introduction

6xxx-series aluminum alloys are widely used for automotive plates due to their high strength-to-weight ratio and good formability. For aluminum sheet and plate products, iron is the most common (and detrimental) impurity, as it forms hard intermetallic compounds that are harmful to the mechanical performance of manufactured parts (such as those shown in Figure 1).

Figure 1. Car frames and body panels are often produced with advanced aluminum alloys.

Microcracks associated with an iron-rich intermetallic particle are shown in Figure 2. These endogenous microcracks may expand as the material is deformed into its final shape, leading to larger material fractures. Scanning electron microscopy (SEM) was used to acquire Figure 2 through a combination of backscattered electron (BSE) and secondary electron (SE) signals. The BSE signal highlights compositional contrast while the SE signal shows sample topography, including features such as cracks or voids.

Thermomechanical processing and alloying impact the formation of these iron-rich intermetallic particles and may be optimized to minimize the risk of microcracking. This application note discusses the effect of nickel microalloying on the size, distribution, and composition of iron-rich intermetallic particles in a 6xxx-series aluminum alloy.

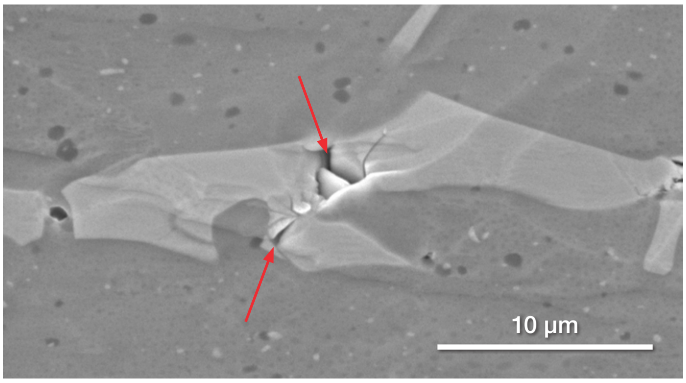


Figure 2. SEM image of an aluminum matrix. Arrows indicate microcracks associated with the bright Fe-rich particles. Dark Mg-Si intermetallic particles are also visible. ***Sample courtesy of the University of Science and Technology Beijing.***

Methods and results

Two batches of 6xxx-series aluminum (trial and baseline) were processed into 1-mm thick sheets through casting and rolling. The applied thermomechanical processing steps are shown in Figure 3, and the base composition is shown in Table 1. Note that the trial composition includes an additional 0.03% nickel. Longitudinal cross-sections were polished with broad ion beam (argon) milling. Manual imaging and automated particle analysis were performed with the Thermo Scientific™ Phenom™ ParticleX™ Steel Desktop SEM.

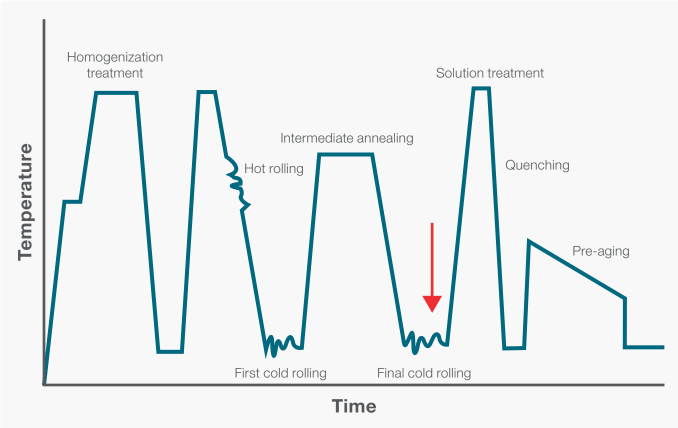


Figure 3. Schematic illustration of the thermomechanical processing of aluminum. The arrow indicates the stage at which samples were taken for analysis.

Mg	Si	Cu	Mn	Fe	Al
0.9	0.7	0.2	0.5	0.4	~97 (Bal.)

Table 1. Base composition of the 6xxx-series alloy by weight percent (wt%).

Manual BSE imaging (Figure 4) provided an overview of the iron-rich particle distribution in the trial and baseline samples. The nickel-containing sample (Figure 4A) appears to have more bright intermetallic particles. Manual spot analysis with energy dispersive spectroscopy (EDS) confirmed the presence of iron in these particles; nickel was also identified in the trial-sample particles. While this spot analysis provided a qualitative assessment of particle composition, statistically relevant data would take an excessive amount of time to collect with this kind of manual approach.

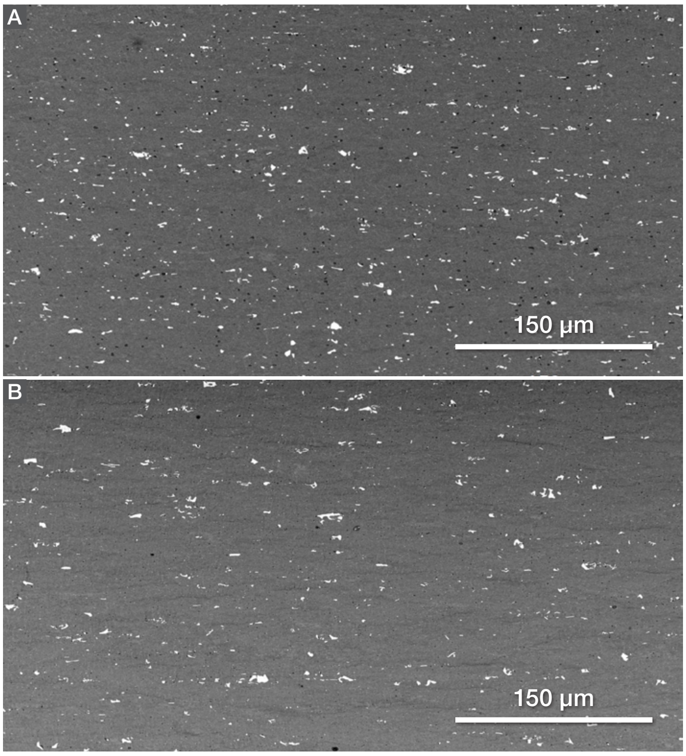


Figure 4. BSE images showing bright, iron-rich intermetallic particles distributed in the aluminum matrix of the trial (A) and baseline (B) samples.

Quantifying intermetallic particles with automated SEM

The Phenom ParticleX Steel Desktop SEM offers fast, straightforward, and automated particle analysis that addresses the need for quick, statistically relevant data. This automated workflow drives the SEM to find and analyze particles that are brighter (or darker) than the base metal. In this case, the instrument scanned a 3 mm^2 area for particles larger than 1 µm. The size, shape, composition, and a BSE image were recorded for every particle identified. This data can subsequently be classified according to any of these recorded variables. In this example, particles with elevated nickel (over 15 at%) were classified as “Ni-containing” in Perception Reporter Software, which allows you to create customized particle tables, histograms, or ternary diagrams.

Figure 5 shows particle size distributions for both samples. With the addition of nickel, the number of particles over 1 µm increased more than +17%. The average particle sizes for the trial and baseline samples are 2.56 and 3.15 µm, respectively. In the trial sample, more particles under 3 µm were formed, and the population of particles over 3 µm decreased. The maximum also moved from 2–3 µm to 1–2 µm with the addition of nickel. These changes indicate that nickel affects the size distribution of the iron-rich phase in the alloy, resulting in the formation of smaller intermetallic particles.

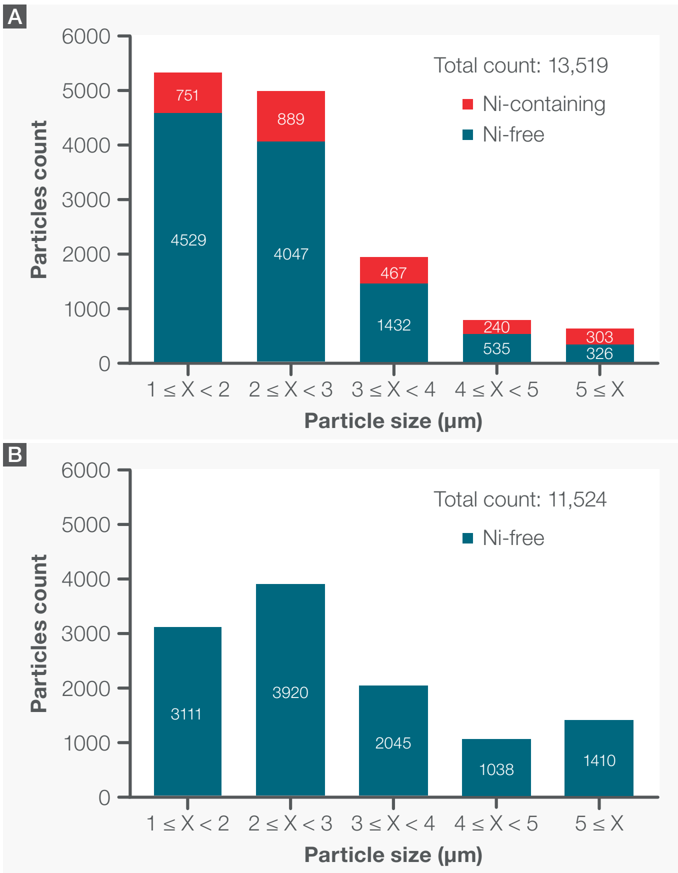


Figure 5. Particle size histograms for the trial (A) and baseline (B) samples.

The ternary diagrams in Figure 6 represent all intermetallic particles observed in the trial and baseline samples. Both have Fe-Si containing particles, but the trial sample shows a second population that contains nickel. Note that, although all these intermetallic particles do contain aluminum, the composition plots in Figure 6 do not take aluminum into account. The normalized composition of the nickel-containing particles ranges from 15% to 75% nickel (at%). Due to the higher melting points of iron and nickel (compared to aluminum), these intermetallic particles are expected to form early during solidification, where segregation may assist in their formation.

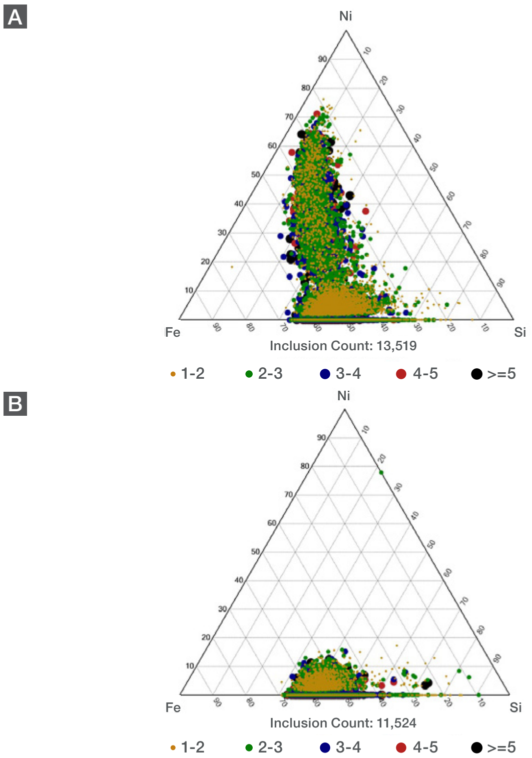


Figure 6. Fe-Si-Ni ternary diagrams of the intermetallic particles in the trial (A) and baseline (B) samples.

Particle Inspector Software for the Phenom ParticleX Steel Desktop SEM permits easy access to recorded information about every particle. Data can be sorted according to each parameter (size, shape, classification, composition, etc.) in order to find the exact particle of interest. Figure 7 shows a sample of this automatically captured data for a nickel-containing particle, including an image, a particle mask, and the quantified composition.

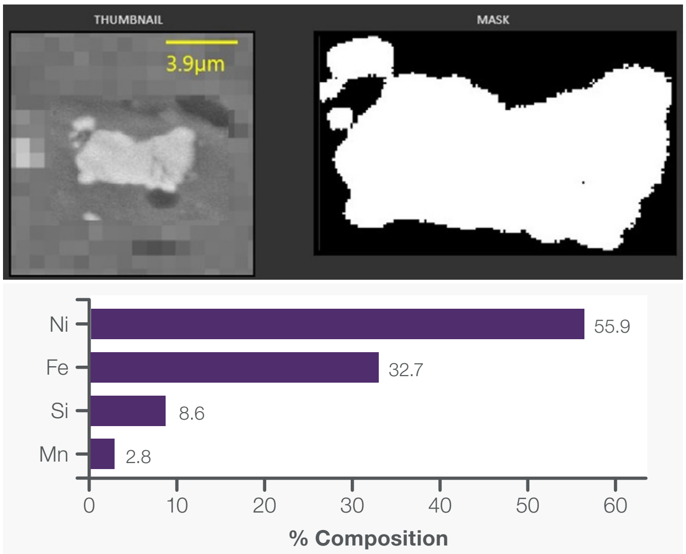


Figure 7. Snapshot of Particle Inspector Software showing the image, mask, and composition for a single nickel-containing particle.

Conclusions

In this application note, iron- and nickel-rich intermetallic compounds were initially identified with manual SEM-EDS analysis, and their particle size and composition distributions were subsequently quantified with the automated workflow of the Phenom ParticleX Steel Desktop SEM. A trial sample, with 0.03 wt% nickel microalloying, was shown to have increased amounts of intermetallic compounds with reduced average particle sizes. The efficiency of the automated workflow made it easy to distinguish the change in particle population for the trial material. Reducing the size of intermetallic particles may improve the fracture mechanics of 6xxx-series aluminum alloys.

Phenom ParticleX analysis of aluminum

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