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Characterization of Rolled and Recrystallized High-Purity Nickel Using EBSP

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As-received, cold rolled and isothermally-recrystallized textures of high purity nickel were studied using electron back-scattered diffraction patterns (EBSP) in a scanning electron microscope (SEM). The evolution of the recrystallization texture was investigated as a function of annealing temperature.

99.995% purity polycrystalline nickel, purchased received from Alfa Aesar, was rolled to either 90 %, 95 % or 98 % thickness reduction at either 293 K or 77 K. The rolled specimens were isothermally annealed in air at their primary recrystallization temperatures and higher temperatures up to 1273 K for 1 h. For SEM/EBSP examination, specimens were mechanically polished, followed by electro-polishing in 10% sulfuric acid in methanol, at a voltage of ~14V, a current density of ~65mA/cm^2 and a temperature < 273 K.

SEM/EBSP study of the as-received nickel showed a strong fiber texture with both <111>//RD and <100>//RD, which is characteristic of extrusion. All the subsequently rolled specimens showed textures typical of a rolled low stacking fault energy f.c.c. metal, see figure 1. A texture composed of both cube and remnant rolling components was observed in the nickel that was 90% cold rolled at 293 K followed by primarily recrystallization at 673 K for 1 h, while a sharp cube texture was observed in nickel 98% cold rolled at 293 K and primarily recrystallized at 658 K for 1 h, see figure 2. In other words, increasing the rolling reduction sharpened the primary-recrystallized, cube texture. Figure 3 shows the {100} pole figures of nickel rolled to 98 % thickness reduction at 77 K or 293 K, followed by primary recrystallization at 643 K and 673 K, respectively. It can be seen that decreasing the rolling temperature does not sharpen the primary-recrystallized cube texture. Abnormal grain growth was found after annealing the rolled specimens at 873 K for 1 h. Therefore, in order to observe the texture evolution after secondary recrystallization, the rolled specimens were annealed at 1273 K for 1 hour. As shown by the pole figure in figure 4, a strong {124}<211> texture was observed, which was also found in our prior study [1] as the preferred orientation for secondary recrystallization in cold rolled nickel. The orientation map in figure 4 shows that the {124}<211> oriented grains comprise about 73% of the total investigated area and, a very small fraction of cube oriented grains still exist in the microstructure.

In conclusion, increasing the thickness reduction during rolling sharpens the primarily recrystallized cube texture in high purity nickel while decreasing the rolling temperature does not. {124}<211> was the preferred orientation for secondary recrystallization in nickel.

References:

[1] Baker I. and Li J., An EBSP study of Isothermally-annealed Cold-rolled Nickel. Microscopy Research and Technique, **63** (5): 289-297, 2004.

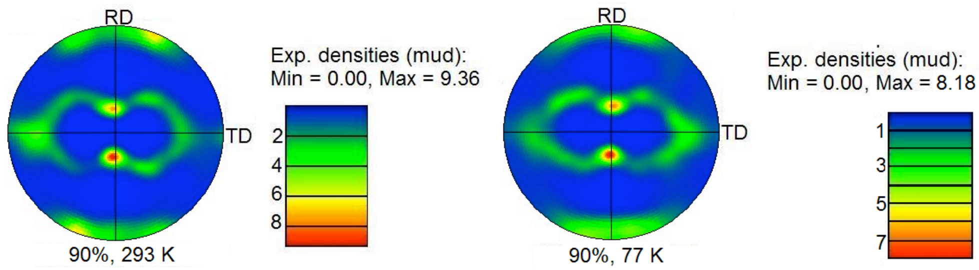

Figure 1. {111} pole figures of nickel specimens 90% and 98% cold rolled at room temperature. The scale shows the probability of the orientation of a particular crystallographic axis.

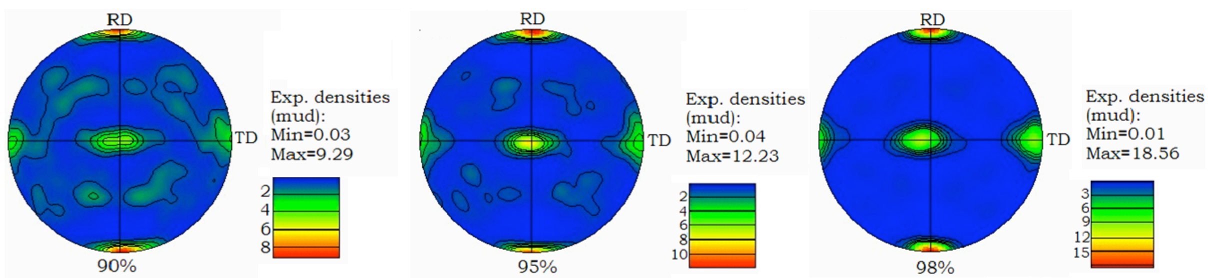

Figure 2. {100} pole figures of nickel cold rolled to 90% 95% and 98% reductions at 293 K, annealed at 673 K, 663 K and 658 K for 1 h, respectively. The scale shows the probability of the orientation of a particular crystallographic axis.

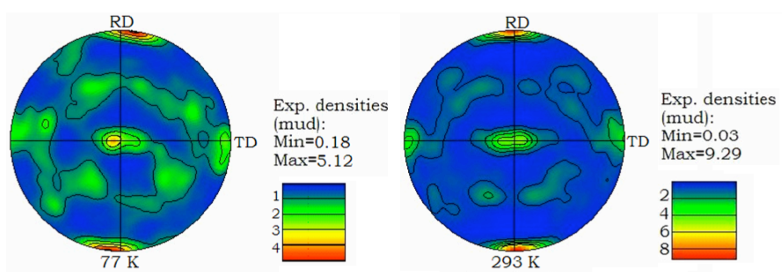

Figure 3. {100} pole figures of nickel 90% rolled at 77 K and 293 K, annealed at 643 K and 673 K, respectively. The scale shows the probability of the orientation of a particular crystallographic axis.

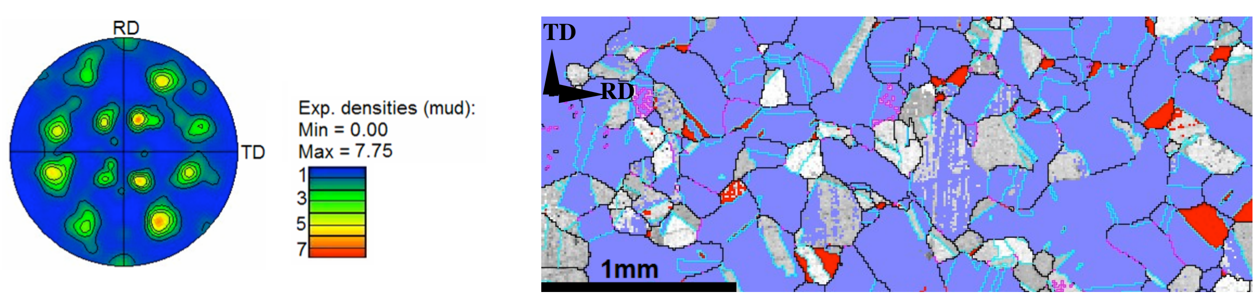

Figure 4. {100} pole figure of nickel 98 % cold rolled at 293 K, annealed at 1273 K for 1 h. The scale shows the probability of the orientation of a particular crystallographic axis, and an orientation map from the same specimen, {124}<211> grains are blue, cube grains are red, Σ 3 boundaries are aqua. Grain boundaries and orientations are set on a 10^{o} misorientation angle.

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