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Formation of annealing twin boundaries in nickel

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The development of annealing twin boundaries in pure nickel was investigated using instantaneous heating in a salt bath and in situ annealing on a heating stage designed for use with electron backscatter diffraction. It was observed that twin grains form to assist in the recrystallization process and that a significantly higher fraction of twin boundaries form when the deformed specimen is heated at a slow rate, regardless of the annealing temperature.

Keywords: Twin boundaries; Recrystallization; Nickel; EBSD

Substantial research has been performed for the evaluation of grain boundary engineered [1–4] materials where a high fraction of "so-called" special boundaries is required. It has been shown that a high concentration of twin boundaries in a material improves the material properties for many different applications [5–7], but there remain somewhat conflicting theories on the development of twin boundaries [8–9]. One field of thought is that they result from growth accidents, grains growing rapidly cause stacking fault accidents to occur, creating an annealing twin (since annealing twins are essentially just regions where the stacking sequence of {1 1 1} planes is interrupted) [10–13]. This theory would suggest that higher annealing temperatures, which cause more rapid recrystallization rates, would result in more twin boundaries. The other theory is that twin boundaries are a result of nucleation via stacking fault creation, which leads to boundaries that are more favorably oriented for growth. In this theory, neighboring grains with similar orientation or low boundary energy tend to be relatively stable, even though one grain may be a growing recrystallization nucleus and the neighbor a deformed grain. When a stacking fault occurs, allowing the recrystallizing grain to form a new orientation, the new twin boundary may increase the driving force for growth, but may result in an overall lowering of energy because the new orientation results in a high-mobility grain boundary that can continue growth more rapidly. If this theory is more accurate, it would imply that a higher fraction of twin boundaries would develop at lower annealing temperatures [14,16].

This study was designed to gain a more accurate understanding of the development of annealing twins in nickel using a single-step process involving high deformation and annealing at different temperatures to complete recrystallization. Research performed by Kumar et al. for a similar process with Cu had inconclusive results regarding the effect of temperature on twin density [15]. Other work on Cu shows a tendency towards higher twin densities when annealed at lower temperatures, which could suggest that the temperatures used by Kumar et al. were too high to see definite results [16]. The most common process for creating a high twin density is called strain annealing, and involves several steps of small deformations followed by annealing at a high temperature. Many studies have already been done on the effectiveness of single-step processes vs. that of multi-step processes [17–20], with differing conclusions. Some studies have also shown improvements in material properties after a single-step process [17,21,22], but the question remains whether or not sufficient improvements in material properties can be made in a single step. If a better understanding can be reached of the mechanism causing annealing twins, a cheaper preparation process can be identified, be it a single-step process or a more efficient multi-step one.

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Specimens were thermomechanically treated using a process involving deformation and annealing of the specimens. The initial cylindrical nickel samples (99.9% pure) measured 23 mm in diameter and had a length of 30 mm. A hydraulic press was used to deform the cylinders laterally and to alter any existing grain structure present in the metal. The sample was further deformed by redundant forging to 25% compressive engineering strain in each of three orthogonal directions. Slices of about 1 mm thickness were cut from the middle of the deformed specimen. All pieces to be annealed were cut from these 1 mm thick cross-sections of the same deformed sample. Only one deformed sample was used to avoid the possibility of differences in the initial stored energy, and the samples were all taken from interior portions of the deformed billet in an attempt to avoid regions of large heterogeneity in the sample. For each annealing temperature and time, at least two samples were taken to ensure repeatability of the results observed.

A control specimen was mounted in a Bakelite mold and prepared using mechanical grinding and polishing, with final polishing steps of 0.1 μm alumina at approximately 300 rpm and 0.02 μm colloidal silica suspension on a vibratory polisher for 12–24 h. The specimen was examined with electron backscatter diffraction (EBSD) analysis to determine the uniformity of strain produced during the deformation process. Results showed that the original grain boundary structure had been significantly deformed over the entire specimen, with an apparently uniform strain distribution. Small specimens were cut from the 1 mm thick sections and annealed in a salt bath at temperatures of 600, 650, 700 and 750 °C. The samples were then rapidly cooled by quenching in water immediately after annealing to ensure that the time at high temperature was accurately known. These samples were then mounted in Bakelite molds for polishing as outlined above. As the mounting press temperature is much lower than that of the temperatures at which the specimens were annealed (and a small fraction of the homologous melting temperature), it is assumed that there was no structural evolution during mounting. The samples were examined using EBSD analysis to determine the fraction recrystallized. The recrystallized fraction was quantified automatically using EBSD data based on a grain size definition of 10 pixels, a misorientation angle definition of 5° and a criterion that assumes the grains are recrystallized if the orientation spread is 1° or less. The average grain diameter and the fraction of twin boundaries were also determined for each specimen. All EBSD data were adequate for analysis, with no clean-up or data altering procedures other than ignoring any points for which the confidence index was less than 0.2 and imposing a grain confidence index standardization based on the grain definition given previously. All data sets had over 97% high confidence data and included more than 500 grains per specimen, so the orientation and boundary statistics were reasonable for each data set.

Several samples were also studied in situ to analyze local structure evolution. These samples were prepared in a similar fashion to those annealed in the furnace but with more specific dimensions to fit the heating stage of 5 mm × 7 mm × 1 mm. (The stage was designed specifically for EBSD analysis and was obtained from TSL Solutions KK.) They were then raised to temperatures of 725, 700, 675 and 600 °C at a heating rate of 5 °C s^{-1}. After the specimens had reached the annealing temperature, EBSD scans were done every 2 min until complete recrystallization had occurred. Since the heating method was much slower, allowing for more recovery than the salt bath samples, one sample was annealed in air at 700 °C in the furnace to verify that surface analysis (as opposed to bulk analysis) of the material during recrystallization did not change the results for the twin boundary content. As in most in situ studies, the apparent grain size of the in situ structure is larger than that obtained in the specimen interior, but the twin boundary content remained consistent. This indicates that the results obtained are reliable as far as the twin boundary fraction is concerned, even though the grain sizes would be different had the observations been of the specimen interior.

After deformation, samples were annealed in salt baths at 600, 650, 700 and 750 °C for various times to estimate the kinetics of the process. The recrystallized portions of these samples were used to determine average grain size and fraction of twin boundaries for the given annealing temperature. Complete recrystallization was never achieved at an annealing temperature of 600 °C, where, after a period of annealing of 240 h (10 days), the sample had only 65% recrystallization, as seen in Figure 1. Several samples were also annealed in situ at temperatures of 600, 675, 700 and 725 °C. A graph of the annealing time vs. the percent recrystallized for specimens annealed in salt baths at 650, 700 and 750 °C is shown in Figure 1, with possible Avrami curves for each. The error bars show the positions of the maximum and minimum values obtained for each point if only two samples were measured. Where three or more samples were measured, the error bars show the standard deviation from the average value. Complete recrystallization occurred after about 4 h when the structure was annealed at 650 °C, compared to 2 h for the specimens annealed at 700 °C and 0.2 h at 750 °C.

Figure 2 shows the average grain size for each salt bath annealing temperature and each annealing time investigated. The grain sizes reported here include twin grains treated as separate from the parent grains. The grain sizes were obtained from EBSD data using a grain definition of 5° and a minimum size of 10 measurement points per grain. In general, the average grain diameter decreases as the temperature decreases. This could have

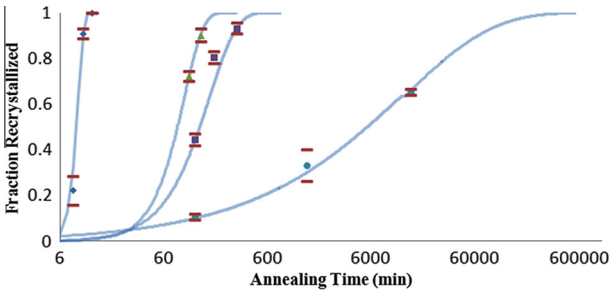

Figure 1. Recrystallization times and superposed Avrami curves for specimens annealed in salt baths at 600, 650, 700 and 750 °C.