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The tensile behavior and deformation microstructure of cryo-rolled and annealed pure nickel

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Abstract

The deformation microstructures of room temperature and cryo-rolled commercially pure nickel to a thickness reduction of 95% were studied by transmission electron microscopy (TEM). Lamellar boundary spacings and boundary misorientations were measured and analyzed. It was found that the only difference between these two microstructures was boundary spacing. After annealed under different annealing conditions, the tensile behaviors of rolled and annealed pure nickel were studied. It was found that the higher the strength after annealing, the lower the ductility. The combination of high strength and high ductility behavior found in copper by cryo-rolling and annealing was not found in the present research.

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1. Introduction

It is a general consensus that an increase in the strength of a metallic material by work hardening will decrease the ductility of the material. The report by Wang et al. [1] of a significant increase in yield strength of pure copper was combined with virtually no loss of ductility by cryo-rolling and annealing, is therefore particular interesting. Wang et al. rolled pure copper at liquid nitrogen temperature to 93% thickness reduction, and found the yield strength was increased nearly six times, however necking occurred right after yield, so that the ductility was lost. However, by annealed the cryo-rolled copper at a proper annealing condition, in this case at 200 °C for 3 min, they found that the yield strength only dropped slightly, but the ductility was increased nearly back to that of the coarse-grained condition. The high strength was attributed to the high density of dislocations in the form of nanoscale networks due to cryo-temperature rolling; and the high ductility was attributed to a bimodal structure generated by annealing. The bimodal structure consisted of a volume fraction of $\sim 25\%$ of 1–3 μ m coarse grains embedded in a very fine matrix structure.

Since room temperature rolling plus annealing cannot lead to this kind of behavior, it must be that the specific microstructure produced by cryo-rolling has to play a important role, unfortunately, Wang et al. [1] reported no detailed information about the cryo-rolled microstructure. It is interested to know the details about the microstructure of cryo-rolled metals. It is also of interest to know whether the above-mentioned behavior could be found in other metals? These are the aims of the present paper: (a) to know whether the behavior reported by Wang et al. [1] in copper can occur in other metals? (b) to know the detailed microstructure of cryo-rolled metals. In the present paper, pure nickel was chosen as the study material.

2. Experimental

Commercially pure Ni 200 with purity of 99.5%, and electrolytic Ni with purity of 99.99%, both in plate form were

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used. Rolling was performed at room and liquid nitrogen temperatures. During rolling, only 10% thickness reduction was rolled by each rolling pass in order to minimize the temperature rise during rolling. The total thickness reduction was 95%; the final specimen thickness was 0.65 and 0.8 mm for Ni 200 and electrolytic Ni, respectively. When rolling at liquid nitrogen temperature, specimen was immersed in liquid nitrogen before and after each rolling pass.

After rolling, specimens were annealed under several annealing conditions, and microhardness testing was used to assess the degree of softening. Specimens with certain frac-



Fig. 1. TEM micrographs showing the deformed microstructure of commercially pure Ni after rolling at: (a) room temperature and (b) liquid nitrogen temperature to a thickness reduction of 95%.

tions of softening were chosen to perform subsequent tensile tests. The tests were performed at room temperature with a strain rate of 7×10^{-4} /s. In order to prepare 3-mm discs for TEM observation, rolled specimens were electro-plated with Ni to a thickness more than 3 mm, after that standard twin-jet electro-polishing was used. Specimens were examined along the transverse direction by a Philips CM200 electron microscope equipped with a Gatan 300 W CCD camera. Lamellar boundary spacings and misorientations were determined. The spacings of lamellar boundaries were measured along the direction perpendicular to the boundaries. The misorientation and the rotation axis of a boundary were measured by analyzing Kikuchi patterns obtained across the boundary. The computer program used to analyze the Kikuchi patterns and to calculate the misorientations and the rotation axes has the trade name of TOCA, which is supplied by TexSEM Laboratories Inc. For each experimental condition, misorientations were obtained from eight different areas, and about 40 misorientations were measured from each area; as for boundaries spacing, 15 different areas were measured, and about 40 spacings were measured from each area. In total, about 300 misorientations and 600 boundary spacings were measured at each rolling temperature.



Fig. 2. The frequency histogram of lamellar boundary spacings of commercially pure Ni rolled at: (a) room temperature and (b) liquid nitrogen temperature, to a thickness reduction of 95%.

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