RELATION BETWEEN MICROHARDNESS AND RECRYSTALLIZATION OF NICKEL

Decrease of hardness is one of the important changes of mechanical properties accompanying polygonization processes and especially recrystallization of metals. In this paper the changes of this property are studied in relation to the recrystallization degree of nickel. Two different grades of purity of the original material were chosen for this investigation: a sheet of Johnson & Matthey's 1) and another sheet of technical nickel 2).

The original nickel sheets 1 mm thick of both of these materials were annealed for one hour in vacuum at $10^{-3}$ torr at 700 °C. The material was then rolled to the thickness of 0.05 mm, so that the total reduction was 95%. The samples so prepared were annealed in a potassium nitrate bath up to the temperature 600 °C. Microhardness measurements were carried out with the microhardness tester PMT-3 with the aid of a Vickers pyramid loaded at 200 g. The resulting microhardness values ($H_{200}$) in Fig. 1 and 2 are the averages of 50 measurements. The precision of measurements may be characterized by the difference between the maximum and minimum values which was of the order of 5% of the average. The samples for the transmission electron microscopy were prepared by electrolytical polishing after Bollmann [1] and for the observation a Siemens Elmiscop I was used at 100 kV. Samples for optical microscopy were electrolytically polished and chemically etched in the concentrated hydrochloric acid.

Microhardness measurements were limited by the material thickness. If the thickness of the samples attains a certain critical limit, the hardness of the underlying plate influences appreciably the results of the measurement. It is evident that the critical thickness increases with decreasing hardness (detailed discussion in [2]). The electrolytic polishing of samples for transmission electron microscopy limits the thickness of original sheet to $\sim$0.05 mm at the most, so that the measured values ($H_{200}$) may really be influenced by the underlying plate. This concerns especially samples in which the hardness decreases due to the recrystallization. However, it is unlikely that the variations of the critical thickness influence appreciably our measurements just in the important parts of the curves, where the changes of microhardness are small. This refers to the upper part of the curve on Fig. 1 describing the first stage of the recrystallization, and to the upper parts of curves on Fig. 2 showing a rapid development of the polygonization in a low purity nickel.

1) Composition: Fe 10 ppm, Si 10 ppm, Al 5 ppm, Ca, Mg, Cu, Au and Ag 1 ppm.
2) Composition: Fe 0.006%, Si 0.05%, Al 0.065%, Co 0.03%, Cu 0.005%, S 0.002% and Zn 0.0068.

On Figure 1 the average microhardness of several samples isochronically annealed is plotted as a function of the annealing temperature. In this case the samples were not previously polished and etched, so that the character of structure in the place of indentation was not known. Polygonization processes can be observed already above 150°C. When the temperature increases up to 200°C, recrystallization nuclei arise. At temperatures above 200°C both processes occur simultaneously and at 300°C recrystallization is almost finished. Among new grains only isolated remainders of polygonized material can be found.

![Fig. 1. The dependence of microhardness of 99.997% nickel which was deformed by 95% on the temperature during 1 hour annealing. The beginning of recrystallization is denoted by the arrow.](image)

Figure 2 represents again the microhardness during polygonization and recrystallization processes. However, samples in this case were electrolytically polished and chemically etched so that it was possible to obtain data separately for different kinds of structure. The upper part of curves in Fig. 2 concerns measurements on polygonized areas and the bottom part corresponds to the recrystallization nuclei and to the new grains of nickel of either purity. The start and the end of recrystallization was determined by transmission electron microscopy. The start is defined as the moment when the first recrystallization nucleus of the diameter 3 μm appears, which is limited by high angle boundaries and has no substructure. The end of recrystallization is the moment of disappearing of the last traces of the polygonized matrix. Figure 3 (see Appendix I, p. 1346a) indicates an example of the character of the recrystallization nucleus magnified 6000×. As the areas of the recrystallization nuclei and of the last traces of the polygonized matrix revealed by the transmission electron microscopy are too small for optical measurements with the microhardness tester, it is not possible to measure the microhardness of the first nuclei and of the last remainders of polygonized matrix. This is demonstrated in Fig. 2.

It is also evident from this figure that polygonization in low purity nickel starts at temperatures by nearly 150°C higher and microhardness changes in polygonized matrix during recrystallization are larger than in the high purity nickel. This is in agreement with the previous work [3] in which it was shown that in the high purity nickel the nucleation begins before a notable polygonization may be observed.