Effect of Rolling Mode on Grain Growth of Electrodeposited Nanocrystalline Nickel

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Abstract. The electrodeposited nanocrystalline nickel samples were deformed by conventional unidirectional rolling, cross rolling and pack rolling, respectively. The effect of rolling mode on grain growth behavior was investigated by x-ray line profile analysis, transmission electron microscopy observation and microhardness measurement. Quantitative analysis indicated that rolling deformation induced grain growth is associated with grain rotation as well as dislocation activity, no matter what the rolling mode we use. Based on grain rotation induced grain growth, cross rolling induced grain growth is the most obvious, followed by conventional unidirectional rolling and pack rolling.

1. Introduction
In recent decades, the grain growth behavior of nanocrystalline materials has drawn much attention because grain size has a fundamental effect on their mechanic behavior, which will be directly related to the practical application. It is universally recognized that the tiny nanograins exhibit very unstable and they inevitably tend to grow up [1-2]. Although a great number of experiments have indicated the growth behavior of nanograins is a complex issue, scientists are trying to slow down or retard their growth tendency [3-4]. There are numerous factors that could affect the grain growth behavior, one of the most common factors is external stress. From the published literature, there are still many differences among various deformation-induced grain growth [5]. It is noted that deformation paths indeed do affect the evolution of microstructures including the grain size [6].

For the electrodeposited nanocrystalline nickel samples with similar initial microstructure, conventional rolling and pack rolling have been conducted at room temperature and different grain growth rates have been found in our previous work [7-8]. However, quantitative and comparative investigation on grain growth during cross rolling is still less. Therefore, in the present effort we extend the rolling deformation mode to cross rolling in the hope to better understand the grain growth behaviors of cold-rolled nanocrystalline nickel.

2. Materials and Methods
Several electrodeposited nanocrystalline nickel sheets with initial thickness of ~0.25mm was first selected by x-ray diffraction (XRD) to ensure the microstructures including average grain size and
texture of the samples in different deformation groups are on the whole as same as possible. And then, the as-received sheet was cut into small rectangular pieces of 8 mm (length) × 6 mm (width) for the subsequent rolling deformation. Three rolling modes were conducted in the present study. The first group is deformed by conventional unidirectional rolling, i.e., the rolling direction is maintained constant during whole rolling process. The second group is deformed by cross rolling, i.e., the rolling direction is rotated once by 90° around the normal direction. The third group is deformed by pack rolling, i.e., two stacked samples were simultaneously rolled by conventional mode. For simplicity, the deformed samples were obtained by the above three rolling modes were afterward referred to as the UR, CR and PR sample, respectively. The nominal rolling strain for these above rolled samples was determined according to Ref. [7-8].

In order to obtain the overall information, the microstructures of the samples were studied by XRD using a Rigaku D/MAX2500PC diffractometer with Cu-Kα radiation, operating in a fixed-time step scan mode. Quantitative microstructure analysis was determined by x-ray line profile analysis [7]. The corresponding mechanical response was determined by micro-Vickers hardness using a dwell load of 0.98 N and a dwell time of 10 s. The final deformed samples were thinned and made into transmission electron microscope (TEM) samples for intuitive observation.

3. Results and discussion

Figure 1 shows the evolution of average grain size of the samples by different rolling modes. Obviously, the grain growth rate is different. The growth rate of the deformed PR sample is the slowest, which is consistent with our previous finding that pack rolling seems to be able to inhibit the grain growth to some extent [8]. However, the growth rate of the deformed CR samples is the fastest, which may be attributed to the role of multi-directional stress. Under the multi-directional stress, it is very likely that more grains could start to rotate, and then grain coalescence will occur spontaneously based on grain rotation as the main mechanism of grain growth [9].

![Figure 1. Grain size evolution of samples deformed by different rolling modes.](image)

Figure 2 shows the ratio of the (111) and (200) diffraction intensities ($I_{111}/I_{200}$) of samples during rolling deformation. The values of $I_{111}/I_{200}$ can indirectly reflect the degree of grain rotation [10]. All of the three samples show that for the most part the values of $I_{111}/I_{200}$ decreases with the increasing nominal rolling strain. This also means typical rolling texture has formed no matter which rolling mode is applied, but there are bound to be differences in intensity. From another point of view, grain rotation in the CR sample seems to be more activity, compared with the two other samples.
Figure 2. The intensity ratio of the (111) and (200) peaks ($I_{111}/I_{200}$) of samples during rolling deformation with different nominal rolling strains.

Figure 3 shows the Vickers microhardness evolution for the rolled samples with various strains. An obvious increase in microhardness is observed, but the increasing degree of the microhardness for the PR sample is significantly lower than that for the CR and UR samples. Besides, it must be noted that the microhardness of the CR sample appears to decrease in the late stage of deformation. Such phenomenon is due to the grain growth. In the previous investigation, it has been found that the density of crystal defects in the deformed microstructure will tend to saturation with the continue of rolling deformation, and further increase of the grain size will dominate the decrease of the microhardness [7, 10]. Overall, there is a good correlation between the increment of the microhardness and the grain size, indicating that the above grain growth behavior is mainly induced by deformation stress.

Figure 4 shows typical TEM results of the samples after final rolling deformation. Grain growth phenomenon is revealed by the bright-field images. Furthermore, high resolution atomic images of grain interior have also indicated a large quantity of deformation defects such as dislocations, stacking faults and deformation twins. Based on the statistical analysis, the results of grain size and dislocation density can support the above mentioned XRD analysis and microhardness measurement. For any rolling deformation mode, the deformation induced grain growth is still associated with grain rotation as well as dislocation activity [11].
4. Conclusion

Three types of rolling modes, including conventional unidirectional rolling, cross rolling and pack rolling, were conducted on the electrodeposited nanocrystalline nickel samples. Comparative study on grain growth was performed by x-ray diffraction, transmission electron microscope as well as Vickers microhardness measurement. The results have implied that rolling deformation induced grain growth is still associated with grain rotation and dislocation activity, no matter what the rolling mode is. Based on the mechanism of grain rotation induced grain growth, the growth rate of the pack rolled sample is the slowest, while that of the cross rolled sample is the fastest.

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