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Internal Friction of Bend-Deformed Nanocrystalline Nickel by Mechanical Spectroscopy *

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Internal friction of nanocrystalline nickel is investigated by mechanical spectroscopy from 360 K to 120 K. Two relaxation peaks are found when nanocrystalline nickel is bent up to 10% strain at room temperature and fast cooling. However, these two peaks disappear when the sample is annealed at room temperature in vacuum for ten days. The occurrence and disappearance of the two relaxation peaks can be explained by the interactions of partial dislocations and point defects in nanocrystalline materials.

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Much attention has been paid to deformation behaviour of nanocrystalline (NC) materials.[1–4] For example, molecular dynamics simulations predicted that dislocation mediated plasticity transited to grain boundary medicated plasticity, such as grain boundary sliding and grain rotation.[5] Experiments have discovered that grain boundary sliding and grain rotation did exist during the deformation of NC nickel (10 nm).[6] However, dislocations and even dislocation pile-up were observed in NC copper (23 nm) and nickel (24 nm) although conventional Frank–Read sources ceased to operate in NC materials,[7–9] which means that dislocation might still affect the deformation of NC materials. The internal friction measurement is an effective way to investigate the deformation behaviour and microstructure of materials.[10–14] Although the internal friction studies of NC materials are of great interest, there are only a few studies on the internal friction of NC Pd,[15] Al,[16–18] Cu,[19] Au,[20] and Ni.[21,22] However, to the best of our knowledge, no research work has been undertaken on the internal friction behaviour of NC materials after deformation. In this Letter, we investigate the mechanical spectroscopy of NC nickel after deformation in order to examine whether there is dislocation-related relaxation peaks at low temperature and give possible explanations.

The electrodeposited NC Ni was purchased from Goodfellow Inc. Figure 1 shows the room-temperature x-ray diffraction (XRD) result of the as-received NC Ni. The two peaks correspond to (111) and (200) peaks, respectively. The average grain size is about 18 nm according to the Scherrer formula. The internal friction Q^{-1} and resonant frequency of the material are measured with frequency modulation acoustic attenuation (FMAA-I) equipment made by the University of Science and Technology of China. Before the testing, the sample ($12 \times 1.5 \times 0.1\,\mathrm{mm}^3$) is bent until the strain reaches 10% and then it is flattened back to rectangular shape. The measurement is performed under the vacuum of 10^{-2} Torr. The sample is fixed on the electrode to form a capacitance. Liquid nitrogen is used to cool the sample to the temperature about 120 K, and then the sample is heated up to 360 K with the heating rate of about 1 K/min. In the second and third runnings, the sample is heated up to 360 K firstly and kept at this temperature for five hours, and then the performances in the first running are repeated. The fourth running is performed when the sample is annealed at room temperature in vacuum for ten days with the same procedure as the first running.

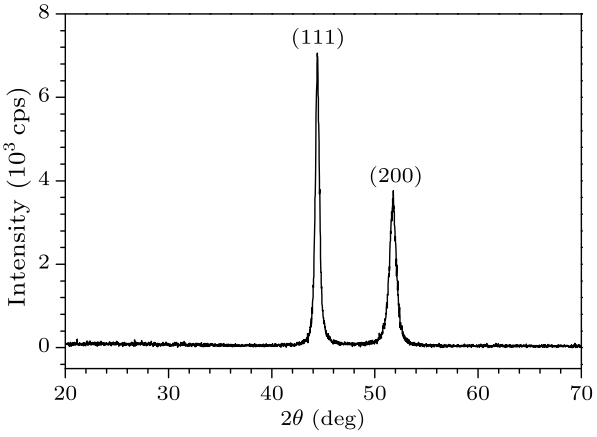

Fig. 1. XRD pattern of the electrodeposited NC Ni.

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Figures 2 and 3 show the temperature-dependent internal friction Q^{-1} and resonant frequency of NC Ni sample. The complex Young's modulus $Y*(\omega)$ can be induced from the change of the resonant frequency of the system. $Y*(\omega) = Y'(\omega)+iY''(\omega)$, where $Y'(\omega)$ and $Y''(\omega)$ are the real and imaginary parts of $Y*(\omega)$.[23] The internal friction Q^{-1} was defined as $Q^{-1} = Y''(\omega)/Y'(\omega)$, which represents the elastic energy dissipation during the vibration. In Fig. 2, no relaxation peak is found in the undeformed sample. However, in Fig. 3, two successive peaks are observed not only in the cooling but also in the heating process during the first three cyclings. The large width of the two peaks indicates that these two peaks are relaxation peaks. The temperatures of peak 1 in Figs. 3(a)–3(c) during cooling are 276.10, 283.45, and 277.72 K while those during heating are 296.85, 308.75, and 310.00 K, respectively. At the same time, the temperatures of peak 2 in Figs. 3(a)–3(c) during cooling are 216.32, 228.67, and 232.00 K while those during heating are 228.67, 252.04, 259.00 K, respectively. The temperatures of peaks 1 and 2 during heating are about 26 and 20 K higher than those during cooling, which may result from the different heating/cooling rates where the cooling rate is faster than the heating rate. Similarly, the values of Q^{-1} of peaks 1 and 2 during heating are also larger than those during cooling. It can also be found that the temperatures of peak 1 are about 50–60 K higher than those of peak 2. Moreover, the peak temperatures of peaks 1 and 2 in Figs. 3(b) and 3(c) are higher than those in Fig. 3(a), which indicates that these peaks are thermally activated. Unfortunately, we cannot calculate the activation energies of the peaks because the length of the sample is so small that it is impossible to perform the torsion pendulum experiment. However, the two peaks should be related to the partial dislocations in the deformed sample. The existence of partial dislocations in the deformed sample is understandable since the sample undergoes the severe plastic deformation. However, no peak could be found when the same sample was annealed at room temperature in vacuum for ten days, as shown in Fig. 3(d).

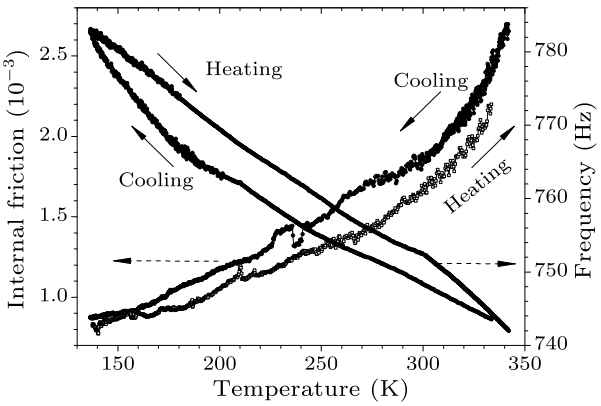

Fig. 2. Internal friction and resonant frequency of undeformed sample.

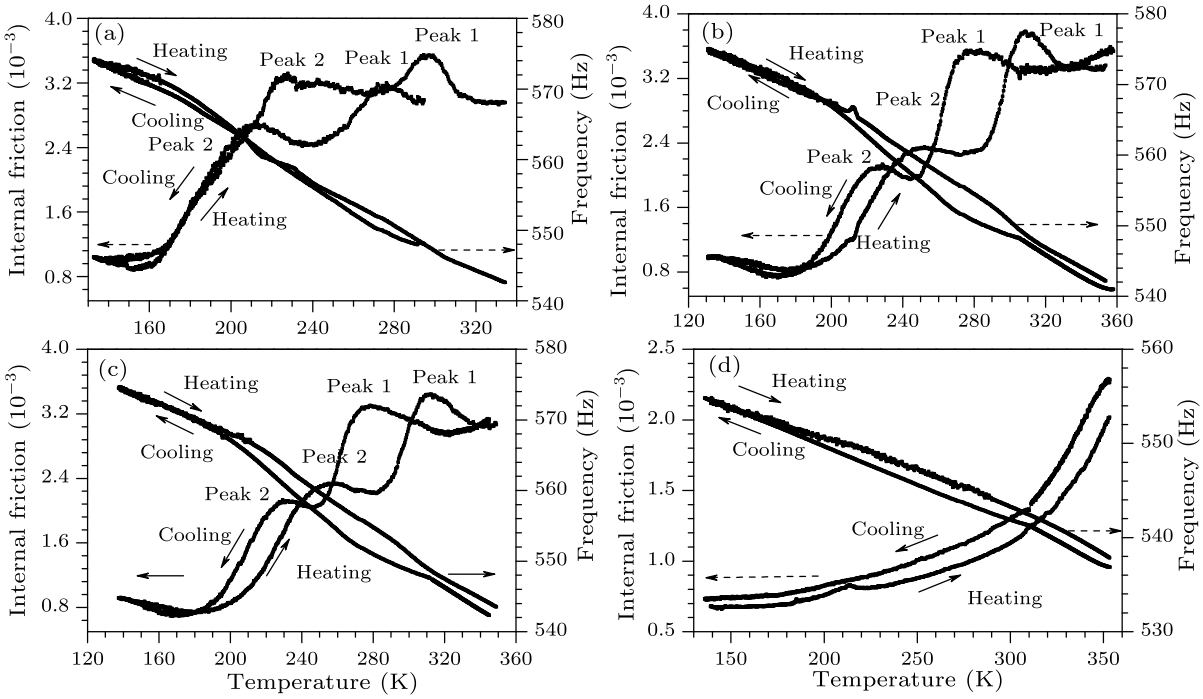

Fig. 3. Internal friction and resonant frequency of deformed sample: (a) cooling/heating in the first running; (b) cooling/heating in the second running; (c) cooling/heating in the third running; and (d) cooling/heating in the fourth running.

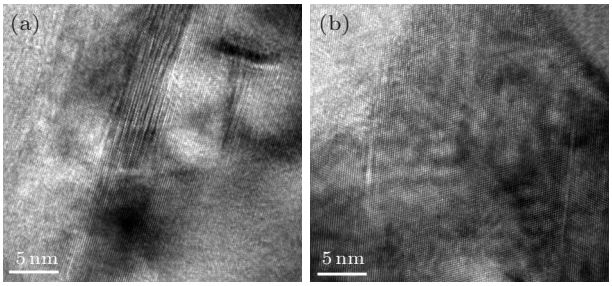

Fig. 4. HRTEM graphs of the nanocrystalline nickel: (a) for the sample before annealing, and (b) for the sample after annealing at room temperature for ten days.

Note that the two peaks occur at fast cooling after room temperature deformation while they disappear after room temperature annealing. These are the characters of Hasiguti peaks, which originate from the interactions of partial dislocations with point defects caused by deformation.[24,25] Although the real process associated with Hasiguti peaks remain uncertainty, a widely accepted model is the thermally assisted de-pinning of partial dislocations and re-pinning of partial dislocations by point defects.[26] During the deformation, partial dislocations and point defects come into being where partial dislocations can be pinned by point defects. De-pinning and re-pinning can take place in the presence of thermal activation, which leads to the occurrence of relaxation peaks. When the deformed sample is annealed at room temperature, the point defect rather than partial dislocation will disappear.[26] In this case, no interactions between partial dislocations and point defects take place and the disappearance of peaks is understandable. Note that the direct observations of the interactions of point defects and partial dislocations are very difficult. However, the microstructure observations of the deformed sample may be helpful to understand the occurrence and disappearance of the two relaxation peaks. Thus, the microstructures of the deformed sample before and after annealing are studied by high-resolution transmission electron microscopy (HRTEM, FEI-TECNAI-F20) operating at 200 kV. Figures 4(a) and 4(b) show the typical HRTEM graphs of the deformed sample before and after annealing. No point defects are observed in Figs. 4(a) and 4(b). However, in Fig. 4(a), a defect like twinning is observed in the sample before annealing and no such defect occurs when the sample is annealed at room temperature for ten days, as shown in Fig. 4(b). These are similar to the behaviour of point defects mentioned above and it is suggested that the interpretation of the two relaxation peaks is reasonable. Definitive observations of point defects and the interactions of point defects and partial dislocations of nanocrystalline nickel requires further HRTEM research.

In summary, the mechanical spectroscopy of NC nickel has been investigated by measuring internal friction from 360 to 120 K. Two relaxation peaks are observed due to the internal friction when the sample is deformed at room temperature and cooled down quickly. However, no relaxation peaks could be found when the deformed sample is annealed at room temperature in vacuum for ten days. These are the characters of Hasiguti peaks of NC materials. The occurrence and disappearance of the two Hasiguti peaks can be explained by the de-pinning/re-pinning of partial dislocations with point defects under thermal activation.

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