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SCIENCE DIRECT®
Materials and Design 26 (2005) 465–468
Materials & Design
www.elsevier.com/locate/matdes

Short Communication

Measurement of microscopic deformation in a CuAlNi single crystal alloy by nanoindentation with a heating stage

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Received 12 January 2004; accepted 12 July 2004
Available online 23 August 2004

Abstract

Thermally induced recovery of nanoindents in a CuAlNi single crystal shape memory alloy was studied by nanoindentation in conjunction with a heating stage. Nanoindents formed by a Berkovich indenter at room temperature were heated to 40, 70 and 100 °C. Partial recovery was observed for the nanoindents. The recovery ratio depended on the heating temperature. Indentation of CuAlNi can induce inelastic deformation via dislocation motion and a stress-induced matensitic transformation. The percentages of dislocation-induced plastic strain would affect the thermal deformation of CuAlNi, because the induced dislocations could stabilize stress-induced martensite plates even when the temperature above austenite finish temperature, A_f. When the applied indentation load is low (less than 10,000 μN), the shape recovery strain is predominant, compared with the dislocation-induced plastic strain. Therefore, the degree of indent recovery in the depth direction, δ_D, is high (about 0.7–0.8 at 100 °C).

Keywords: Microscopic deformation; Nanoindentation; Phase transformation; CuAlNi single crystal shape memory alloy

1. Introduction

Shape memory alloys (SMAs) find applications in microelectromechanical systems (MEMS) and novel medical devices [1–4]. These applications exploit the ability of SMAs to recover large inelastic strains (up to 8% tensile strain for NiTi alloys and 4% for CuAlNi alloys) by heating (shape memory effect-SME) or stress removal (superelasticity-SE). In order to optimally design the future SMAs-based MEMS and medical devices and to improve the performance of a small-scale material system, it requires to have knowledge of microscopic deformation for SMAs. One method used to extract small-scale mechanical properties from materials is nanoindentation. It is known that the indentation of materials creates high stress under diamond indenters that can cause stress-induced phase transformation. Gogotsi et al. [5–7] found that phase transformation occurred in silicon during nanoindentation. A sudden displacement discontinuity (''pop-out'') or a change in slope (''elbow'') of the unloading curve was observed. Gall et al. [8] studied the Vickers microindents in NiTi alloys by transmission electron microscopy (TEM). It was find that microindentation of NiTi alloys above the martensite start temperature, M_s, could induce inelastic deformation via dislocation motion and a stress-induced matensitic transformation. The induced dislocations could stabilize stress-induced martensite plates even when the temperature above austenite finish temperature, A_f. In our study, we examined the thermally induced recovery of nanoindents in a CuAlNi single crystal SMA when heating to 40, 70 and 100 °C. Partial recovery for the nanoindents formed by a Berkovich

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0261-3069/$ - see front matter
doi:10.1016/j.matdes.2004.07.005

indenter was observed. The recovery ratio depended on the heating temperature.

2. Material and methods

A CuAlNi (Cu–14wt%Al–4.12wt%Ni) single crystal SMA was investigated in this research. The transformation temperatures were determined by differential scanning calorimeter (DSC) as: $M_s = 0.5$ °C, $M_f = -3$ °C, $A_s = 26$ °C and $A_f = 36$ °C. The specimen was mechanically polished and finished with 0.05 μm Al_2O_3 suspension.

Nanoindentation tests were performed using Hysitron TriboIndenter® in conjunction with a heating stage. A Berkovich indenter (100–150 nm radius) was used. Before indentation, the specimen was heated to 100 °C for a few minutes and allowed to cool to the room temperature to ensure that no martensite phase existed. At the room temperature, nanoindentation of CuAlNi was conducted. The tested area was marked and the applied maximum indentation loads were 10,000, 5000 and 1000 μN. After in situ imaging of the room-temperature indents, the specimen was subsequently heated to 40, 70 and 100 °C. At each temperature the indents were topographical characterized.

3. Results and discussion

Fig. 1 presents the in situ observation images of nanoindents recovery at temperatures from the room temperature to 100 °C. All the imprint shapes are very sharp before heating in Fig. 1(a).With the increasing temperature, the bottom corner of indents turns blurry (Fig. 1(b) and (c)). At 100 °C, the bottom corner of indents almost completely disappears (Fig. 1(d)). During indentation, a stress-induced martensitic transformation (A → M) occurred [9] in the volume material below the indenter. After the specimen was subsequently heated to 40 °C (>A_f), 70 °C(>A_f) and 100 °C(>A_f), the residual deformation induced by nanoindentation in CuAlNi can be recovered by a reversed phase transformation ($M \rightarrow A$). Cross-section of surface profile measured at different temperatures for the indents at the maximum load of 10,000 μN is shown in Fig. 2. The thermally induced recovery of indents in the depth direction is evident. At high temperatures, indents become shallow

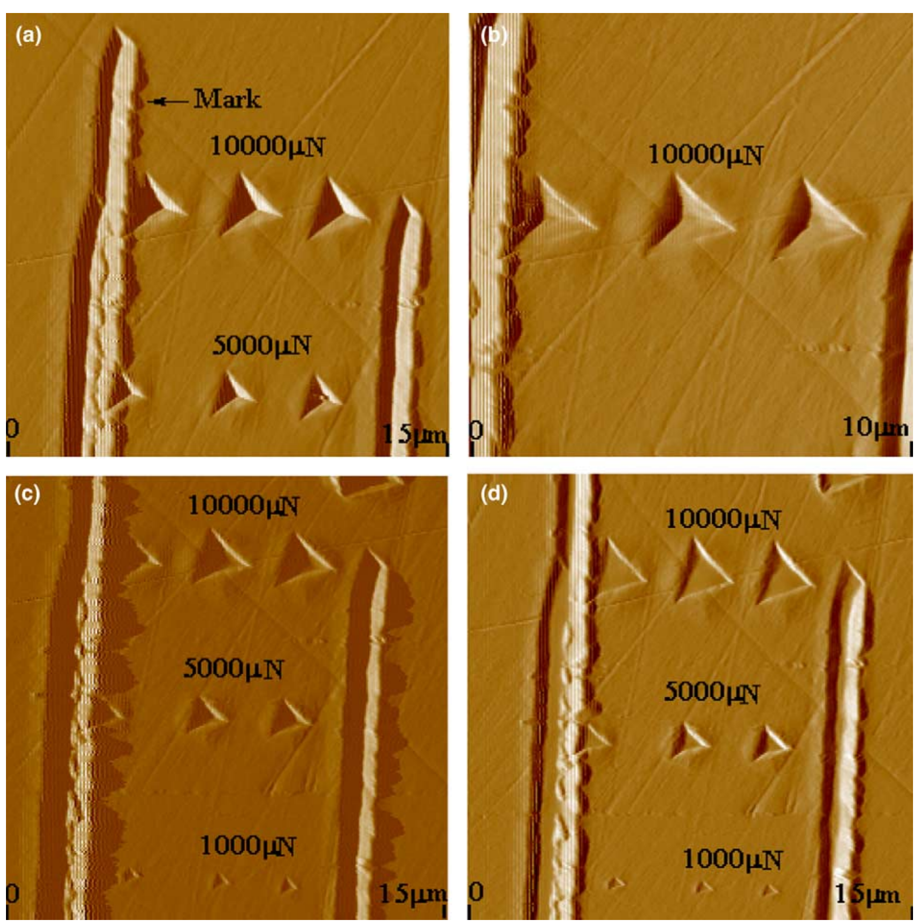

Fig. 1. In situ observation images of the nanoindents at: (a) 25 °C, (b) 40 °C, (c) 70 °C and (d) 100 °C.

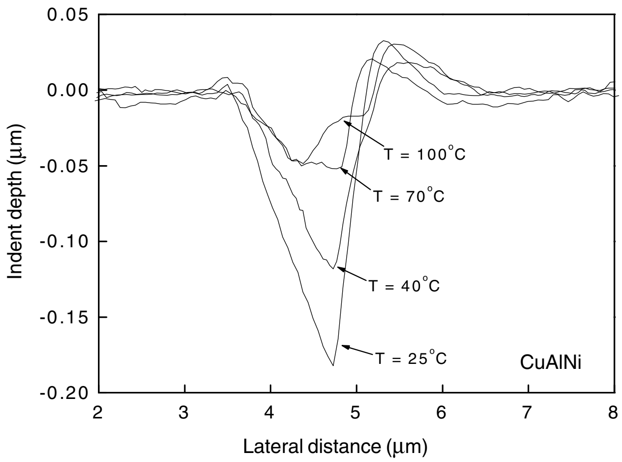

Fig. 2. Cross-section of surface profiles measured at different temperatures for the nanoindents at load of 10,000 μN.

and blunt. Thus, SME exists at the microscopic level and under complex loading conditions. The degree of indent recovery in the depth direction can be determined quantitatively from the surface profile by defining a recovery ratio, δ_D, as [10]:

$$\delta_D = \frac{D_{max}^{T_0} - D_{max}^{T}}{D_{max}^{T_0}}, \quad (1)$$

where D_{max} is the maximum residual indent depth after removal of the load, and the superscripts T_0 and T refer to the room temperature and the heating temperature, respectively. Similarly, the degree of indent recovery in lateral direction, δ_L, is defined as:

$$\delta_L = \frac{L^{T_0} - L^{T}}{L^{T_0}}, \quad (2)$$

where L is the residual indent length along the lateral direction in Fig. 2.

Table 1 summarizes the measured recovery ratios at various loads and temperatures. The recovery ratios appeared to be temperature dependent for temperatures between 40 and 100 °C. At 100 °C, δ_D is about 0.7–0.8 for loads 10,000, 5000 and 1000 μN. In Ni et al.'s study [10], the δ_D for Vickers microindents in NiTi alloys is

Table 1
Recovery ratios for indents at various loads and temperatures

Load (μN)	Temperature (°C)	δ_D	δ_L
10,000	40	0.36	0.10
	70	0.72	0.11
	100	0.75	0.12
5000	40	0.11	0.05
	70	0.55	0.11
	100	0.67	0.16
1000	40	0.09	0.04
	70	0.22	0.07
	100	0.79	0.14

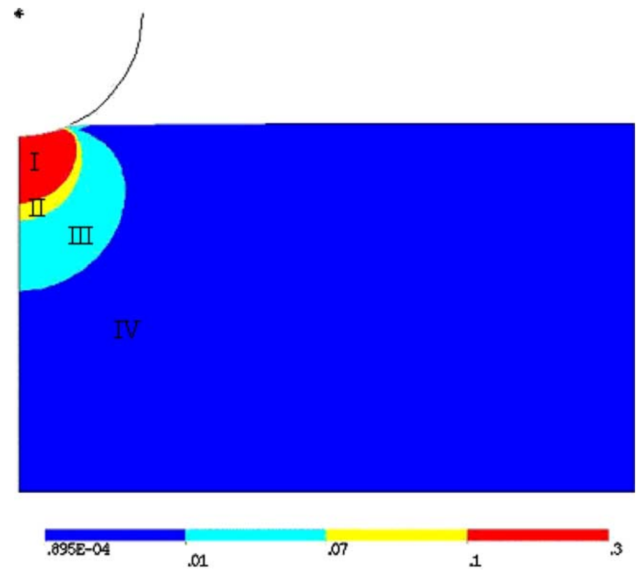

Fig. 3. The spatial effective strain distribution under an indenter in CuAlNi at room temperature.

only about 0.3 at 150 °C at loads between 50 and 2500 mN. The great differences in the recovery behavior between Ni et al.'s and our study possibly lie in the applied indentation load range. The volume material directly under a sharp pyramidal indenter is so highly strained that significant inelastic deformation occurred by dislocation motion as well as twinning mechanisms. The volume dominated by dislocation-induced deformation would not only fail to recover, but could also inhibit shape recovery strain in the underlying materials. Therefore, the recovery of the indents is incomplete and a fraction of the indent impression depth persists following heating. Moreover, when applied loads are very large, the percentage of dislocation-induced plastic strain is higher than that of shape recovery strain. Therefore, δ_D is lower in Ni et al.'s study. In Table 1, it is also found that $\delta_D > \delta_L$ at the same indentation load and temperature. This also has relation to the magnitude and spatial distribution of strain below the indenter. The spatial effective strain distribution under an indenter in CuAlNi at room temperature was simulated by ANSYS 5.7 (shown in Fig. 3). The simulated indenter shape is spherical (100 nm radius) now that the Berkovich indenter is not ideal. The plastically stress-induced martensite phase (Zone I in Fig. 3) occurs right below the indenter and the effective strain is up to 0.3. Adjacent to the plastic zone are elastic martensite phase zone (Zone II) and martensitic transformation zone (Zone III) in which a stress-induced phase transformation ($A \rightarrow M$) occurs. Zone IV is the elastic austenite phase. From Fig. 3 it is found that the widths of Zones II and III in the depth direction are larger than those in the horizontal direction. Therefore, the recovery ratio of indent in the depth direction is larger ($\delta_D > \delta_L$) when heating.

4. Conclusions

Nanoindentation of a CuAlNi single crystal SMA can induce inelastic deformation via dislocation motion and a stress-induced martensitic transformation. The volume dominated by dislocation-induced deformation would not only fail to recover, but could also inhibit shape recovery strain in the underlying materials. Therefore, the recovery of the indents is incomplete and a fraction of the indent impression depth persists following heating. When the applied indentation load is low (less than 10,000 μN), the shape recovery strain is predominant, compared with the dislocation-induced plastic strain. Therefore, the recovery ratio is high (δ_D is about 0.7–0.8 at 100 °C).

Acknowledgements

The research project is supported by the Joint Lab on Microsystems between the Institute of Mechanics, CAS and the Hong Kong University of Science and Technology (HKUST), China under a Grant from HKUST (Project No. CMI00/01.EG02). This work is also supported by the Distinguished Young Scholar Fund of NSFC (Grant No. 10225209), key project from the Chinese Academy of Sciences (Grant No. KJCX2-SW-L2) and National "973" Project (Grant No. G1999033103). The authors gratefully acknowledge Dr. Q.P. Sun of HKUST for providing the sample, and Mr. William C.C. Wong and Mr. Peter K.C. Lee of HKUST for their sincere help in nanoindentation work. The authors also thank Dr. Z.D. Hu of Tongji Univeristy, Shanghai for the simulation work.

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