V-shape in Young's modulus versus strain relationship in shape memory alloys upon mechanical loading

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Abstract

The evolution of Young's modulus in NiTi polycrystalline wires of different initial phases in uniaxial tension was investigated. A V-shape with the bottom at round the hardening start point was found in all experiments. This is a phenomenon that has not yet been reported in the literature. Similar phenomenon was also observed in an austenite CuAlNi single crystal under uniaxial compression although appearing within a much narrower strain range. Optical observation and Wyko interferometer study of the CuAlNi sample surface revealed that the occurrence of V-shape is accompanied by the abrupt reorientation of martensite variant(s). It is concluded that same mechanism should be responsible for the similar phenomenon in polycrystals.
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1. Introduction

At present, more attention is focused on shape memory alloys (SMAs) due to their unique properties [1]. Compared with other smart materials, as actuators, they can provide a larger force over a longer distance. Their thermo–mechanical behavior has been investigated extensively in the past and a vast amount of experiments on SMA single crystals and polycrystals at different temperatures and phases has been reported in the literature.

Generally speaking, there are two types of transformations in SMAs, namely, the phase transformation between the high temperature austenite phase and the low temperature martensite phase and reorientation among martensite variants. The exact behavior of a SMA upon loading is temperature and phase dependent. Normally, the stress versus strain relationship of a SMA at a given temperature can be divided into three regimes as illustrated in Fig. 1. Point *o* to point *a* is elastic deformation, which might be not so apparent at around the

martensite start temperature due to the V-shape phenomenon in the transformation start stress versus temperature relationship [2]. Point *a* to point *b* is the regime dominated by the phase transformation and/or reorientation associated with a very large recoverable strain. The resultant martensite variant(s) is in favor by the applied stress (in single crystals, e.g., [3,4]) or the applied stress and constrains from the neighbouring grains (in polycrystals). Transformation front movement may be observed in this regime [5,6]. Point *b* to point *c* is the hardening regime in which some further transformation occurs together with plastic deformation, in particular in the range close to fracture point *c*.

A typical character of the phase transformation in SMAs is surface relief [1], which can be observed by optical or other microscopes [7,8]. Furthermore, by measuring the surface roughness, one can even quantitatively determine the shape memory effect [9].

It has been known for decades that the Young's modulus of SMAs is phase dependent. The difference in the Young's modulus between austenite and martensite can be as much as a few times in some SMAs [10]. However, upto date the investigation on the evolution of Young's modulus upon me-

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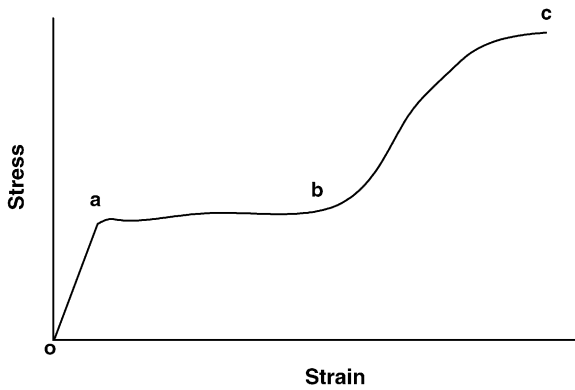 *E-mail address:* mwmhuang@ntu.edu.sg (W.M. Huang).

Fig. 1. Typical stress vs. strain relationship.

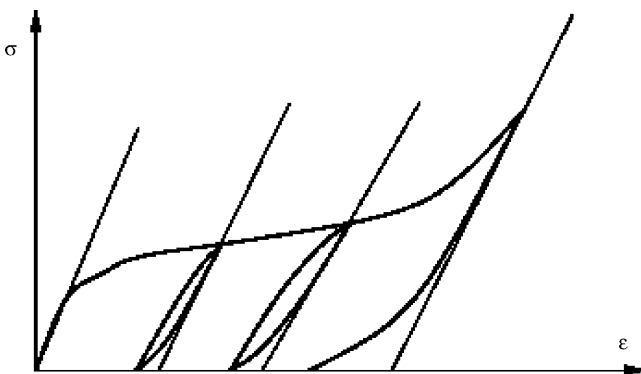
Fig. 2. Determination of Young's modulus [6].

chanical loading is still very much limited. Among other approaches, the Young's modulus can be determined by measuring the slope at the very beginning of unloading path as illustrated in Fig. 2. Huang [6] observed in an initial R-phase NiTi polycrystalline wire that the Young's modulus does not decrease monotonously but increases in the later stage upon uniaxial tension (Fig. 3). However, the test reported by Xiang and Liu [11] on NiTi polycrystalline wires of different initial phases shows no sign of increase at all. In contrast, Huang
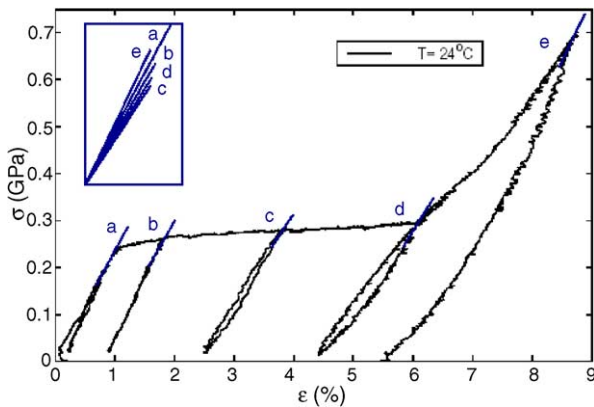

Fig. 3. Stress vs. strain relationship of a NiTi polycrystalline wire under cyclic tension (initially R-phase) and Young's modulus measured at a few points [6].
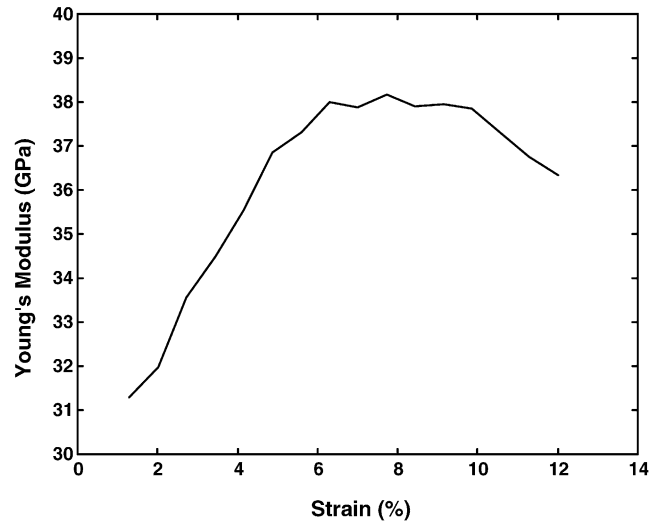

Fig. 4. Young's modulus vs. strain relationship of a NiTi polycrystalline NiTi wire (initially twinned martensite phase) [12].

and Lim [12] reported that the Young's modulus of a martensite NiTi wire increases and then decrease slightly in the very later stage in uniaxial tension (refer to Fig. 4). It appears that there is still no consistent conclusion at present.

In this paper, first we report an experimental study on the evolution of Young's modulus in NiTi polycrystalline wires in uniaxial tension at different temperatures. And then a CuAlNi single crystal was tested under uniaxial compression, in which abrupt reorientation was observed by means of surface observation. This phenomenon seemingly provides a mechanism to explain the V-shape pattern in the Young's modulus versus strain relationship of both materials.

2. Tensile test (NiTi polycrystalline wire)

The raw material was bought from the Shape Memory Applications, INC., USA. It is NiTi polycrystalline wire
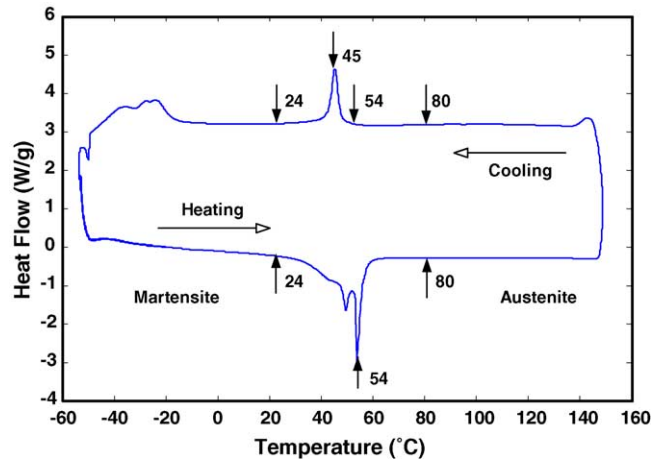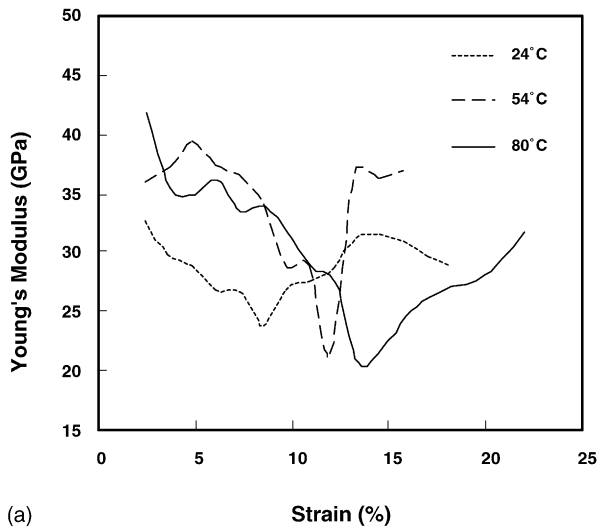

Fig. 5. DSC result of NiTi polycrystalline wires.

with a diameter of 0.55 mm. After heat-treated at 500 °C for 1 h, differential scanning calorimeter (DSC) test was carried out to find the transformation temperatures of this material. The heating/cooling rate was set as 10 °C/min. According to the DSC result, Fig. 5, at 24 °C, which is slightly above the room temperature (about 22 °C), the material is martensite, while at 80 °C it should be austenite.

The samples for testing were cut from the heat-treated wire into about 90 mm in length, while the gauge length in testing was set as 40 mm. A temperature controllable Instron 5569 machine was used for cyclic uniaxial tension tests. Referring to Fig. 5, seven temperatures were chosen for testing, namely, 24, 54 and 80 °C in the heating process, and 80, 54, 45 and 24 °C in the cooling process. In the heating process tests, the samples were pre-heated to

120 °C and then cooled to room temperature followed by heating to the testing temperature. In the cooling process tests, the material was heated to 120 °C and then cooled to the required temperature directly. A small preload of 2 N ($\approx$8.4 MPa) was applied to ensure the wire is straight. The applied loading/unloading rate was 0.04 mm/s (corresponding to a strain rate of 10^{-3} s^{-1}). The loading/unloading cycle was carried out in a 0.50 mm interval until failure (refer to Fig. 7).

The obtained Young's modulus versus strain relationships of all tests are presented in Fig. 6. It is apparent that in general the Young's modulus does not decrease monotonously with the increase of strain but increases in the later stage. A V-shape can be easily identified with the bottom located at round the position, where the hardening starts (refer to Fig. 7 for two typical results). This is a phenomenon different from all those previously reported in the literature.
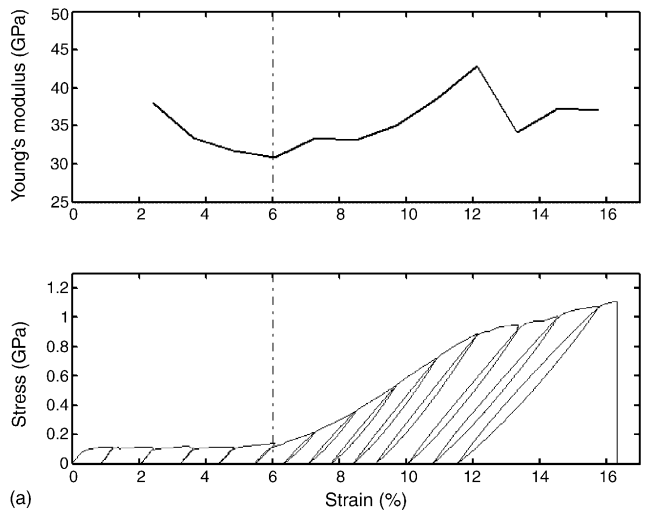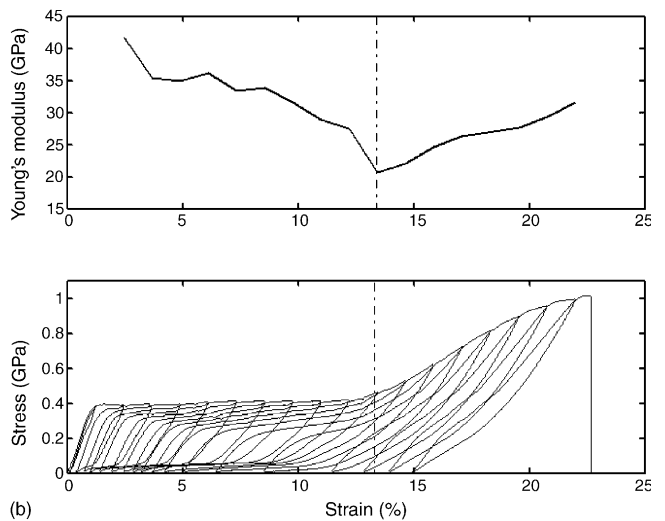

(a)
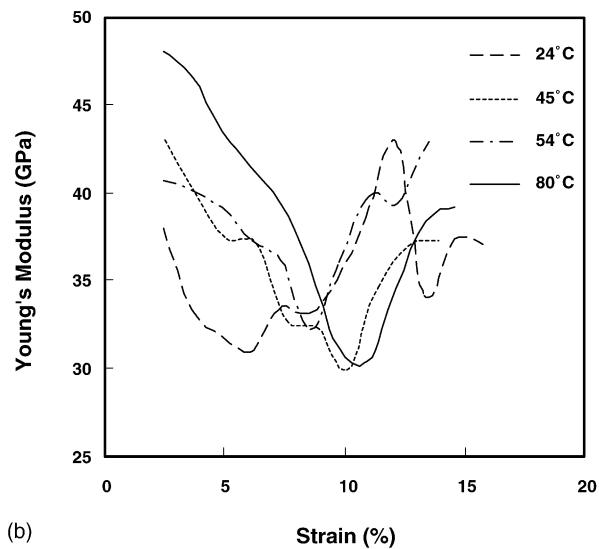
(b)

Fig. 6. Young's modulus vs. strain relationships of NiTi polycrystalline wires in (a) heating process and (b) cooling process.

(a)

(b)

Fig. 7. Typical results of NiTi wires. (a) At 24 °C in cooling process; (b) at 80 °C in heating process.
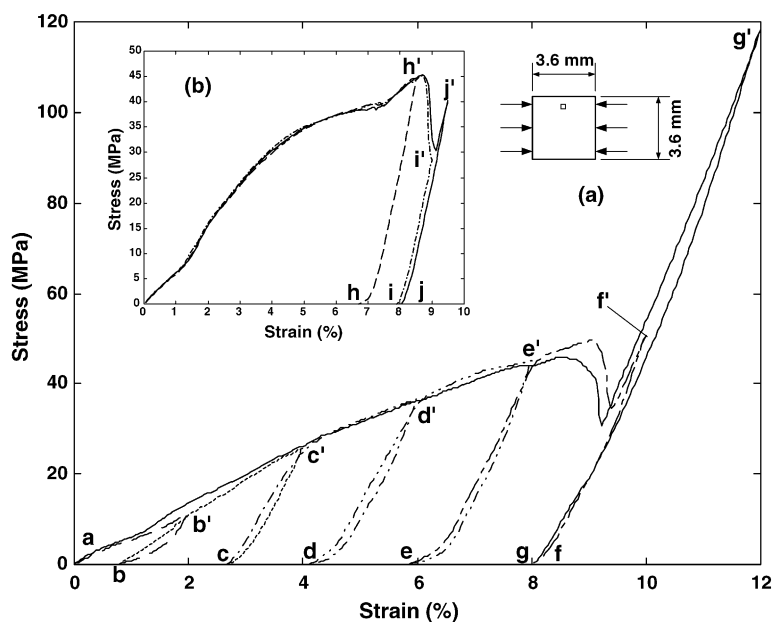
Fig. 8. Stress vs. strain relationship of CuAlNi single crystal.

3. Compression test (CuAlNi single crystal)

The CuAlNi single crystal samples were cut from a self-prepared rod in a diameter of 6 mm. Its composition was identified as $Cu_{81.3}Al_{13.6}Ni_{5.1}$ (wt.%), and the transformation temperatures were determined by DSC test as austenite finish temperature (A_f): 35.5 °C, austenite start temperature (A_s): 20.6 °C, martensite start temperature (M_s): 21.1 °C and martensite finish temperature (M_f): 2.8 °C.

In order to obtain a good surface for later surface study, one side of the sample (1 0 0) was mechanically polished. Note that in the final polishing step, the sample was polished using 1 μm diamond paste at about 80 °C to achieve shiny surface at the austenite phase for easy comparison. The final dimension of the sample was measured as 3.6 mm × 3.6 mm × 3.9 mm (Fig. 8a), where the 3.9 mm direction (not shown) is (1 0 0).

Uniaxial compression test was carried out using an Instron 5565 at room temperature. The force was applied in the direction illustrated in Fig. 8a. Both the loading and unloading rates were set as 3.6×10^{-4} mm/s (corresponding to a strain rate of $10^{-4} s^{-1}$). Before each test the sample was pre-heated to 100 °C. It was found that even after loaded to a 12% strain, the sample was still able to fully recover upon heating to 100 °C. After cooling back to room temperature it should be austenite according to the DSC result.

Note that since no additional measure was taken, the real applied stress may not be perfect uniaxial compression. Apart from that, it is normally very difficult to apply a constant stress state on a bulk single crystal. Hence, similar to these reported in the literature [7], it was expected that the deformation/transformation in our sample might not be uniform everywhere.

In the first test, the sample was loaded to 12% strain and then unloaded. An optical microscope (and in some later tests a Wyko interferometer, which provides more reliable qualitative results) was used to reveal the surface morphology after unloading. Here, only the results around the one marked in Fig. 8a are presented. It is observed (solid line in Fig. 8) that there is an abrupt softening at around 9% strain followed by dramatic hardening. This is anomalous since no previous experimental result reported in the literature shows the similar behavior.

Consequently, cyclic tests were carried out on the same sample (dash-dotted line in Fig. 8). It reveals that the envelope of cyclic test result is very close to the previous one. As we can see in Fig. 9, before testing the surface is as shiny as mirror. Upon loading, it gradually becomes rougher due to the surface relief of the resultant martensite variant(s). Between (e) (8% strain) and (f) (10% strain), where the abrupt softening occurs, there is seemingly a sudden change of pattern in features. In comparison of (f) with (g), which is the result of the previous test after loading to 12% strain, one can see that the surface feature does not change dramatically upon further loading in the hardening regime but the parallel lines are clearer. It reveals the ongoing transformation in the hardening regime. A closer study on the cross-section of the lines after 12% strain by Wyko interferometer shows that these parallel lines are periodical and follow almost an identical pattern.

Additional tests were carried out with focus on the abrupt softening regime. The applied strains were 8.5%
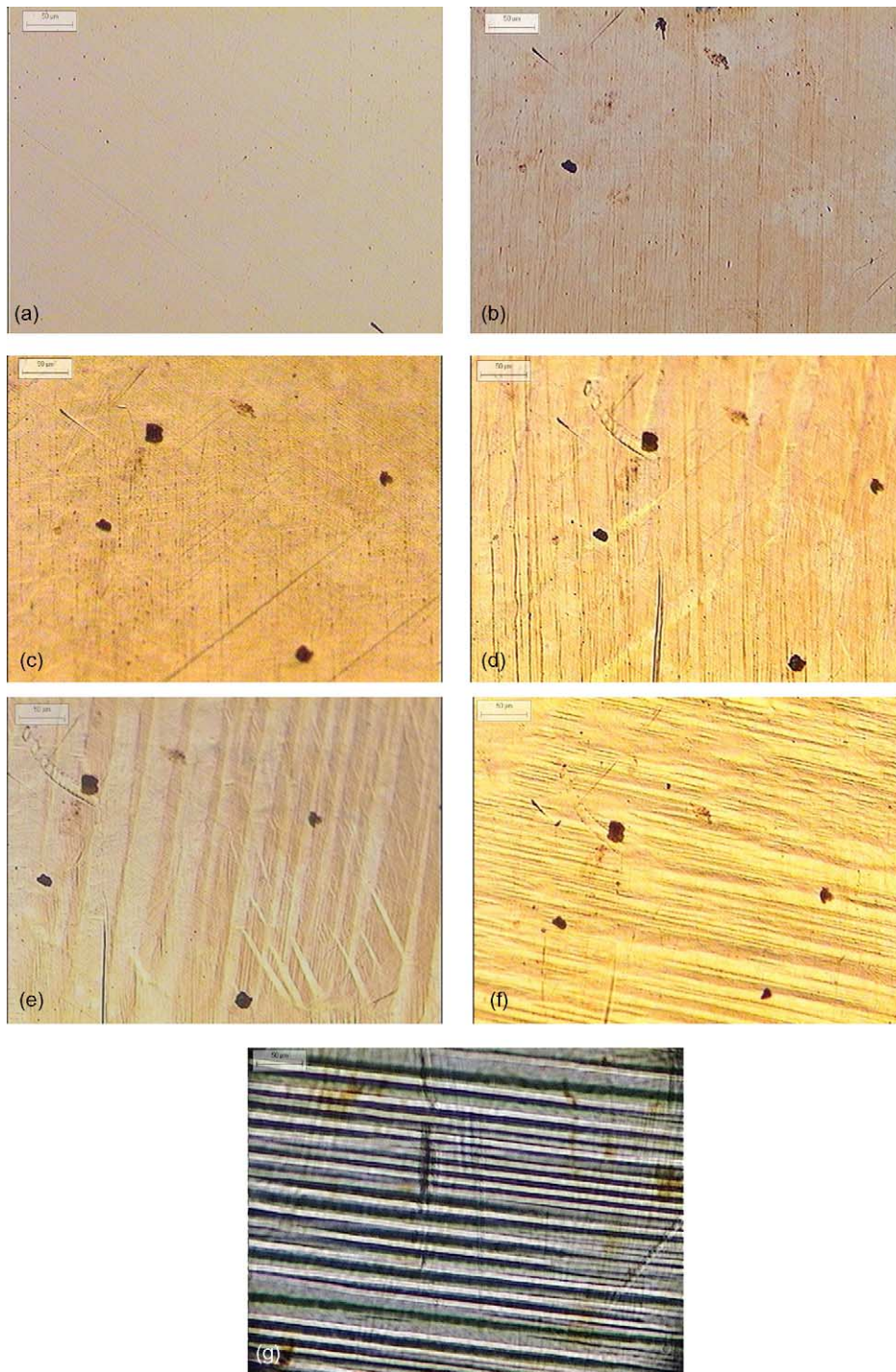
Fig. 9. Optical results. (a) After polishing; (b) 2% strain; (c) 4% strain; (d) 6% strain; (e) 8% strain; (f) 10% strain; and (g) 11% strain. Scale bar is 50 μm. Refer to Fig. 8 for the location where the result was obtained.

(the point just before softening), 9% (the bottom of trough) and 9.5% (after softening). Refer to Fig. 8b for their respective stress versus strain relationships and Fig. 10 for the optical and Wyko (3D) results. We can clearly see, in particular, in the 3D profile that there are two sets of feature lines at 8.5% strain. While at 9% strain one set appears to be dominant at the expense of the other, at 9.5% strain there is not much apparent feature.

The Young's modulus versus strain relationship was obtained from these tests and is plotted in Fig. 11. Again, there is a small V-shape, which is seemingly associated with the abrupt softening in the stress versus strain relationship occurring at around the beginning
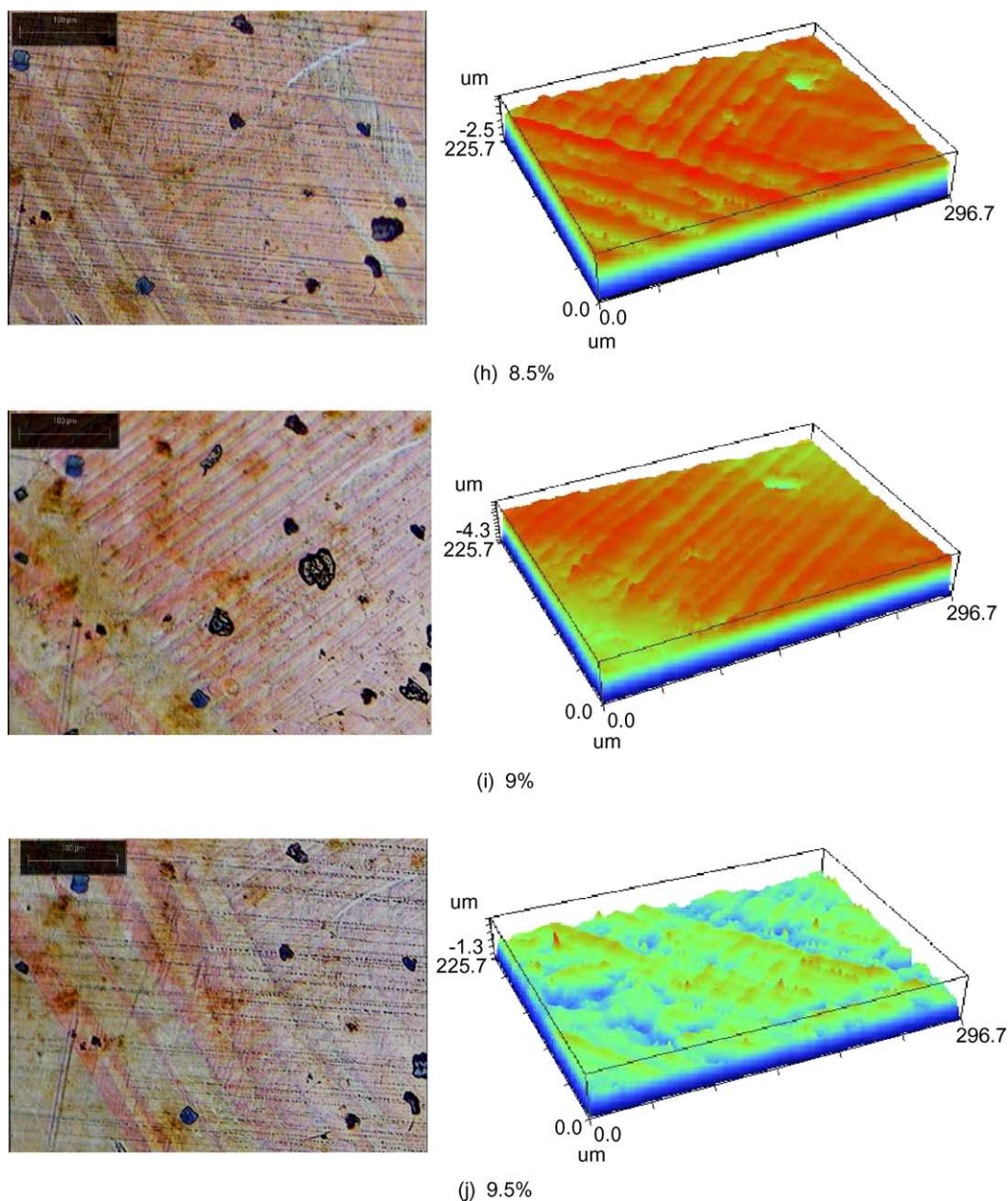
(h) 8.5%

(i) 9%

(j) 9.5%

Fig. 10. Optical pictures (scale bar is 100 μm) and Wyko 3D profiles. Refer to Fig. 8(b) for the location where the result was obtained.

of hardening path in the strain versus stress relationship.

4. Discussion

The V-shape phenomenon in the Young's modulus versus strain relationship was observed in uniaxial tension of NiTi polycrystalline wires of different phases and uniaxial compression of an austenite CuAlNi single crystal. In all tests, the bottom of V-shape was found at round the point where hardening starts.

The surface investigation on CuAlNi single crystal provides clear evidence to show that the stress induced phase transformation from austenite to martensite is not finished in one step but follows a pretty gradual approach, i.e. from austenite to martensite and then reorientation. The hardening in the later stage is the result of growth of particular martensite variant(s), which is in favor by the applied stress state at the expense of others.

As observed in CuAlNi, the abrupt reorientation prior to particular martensite variant(s) becoming dominant proves a good explanation for the mechanism behind the V-shape phenomenon. As shown, the abrupt reorientation is just before hardening occurs. It is also accompanied by an anomalous softening in the strain versus stress relationship. It might be logical to conclude that the reorientation of martensite variant(s) induces a remarkable change of the Young's modulus.
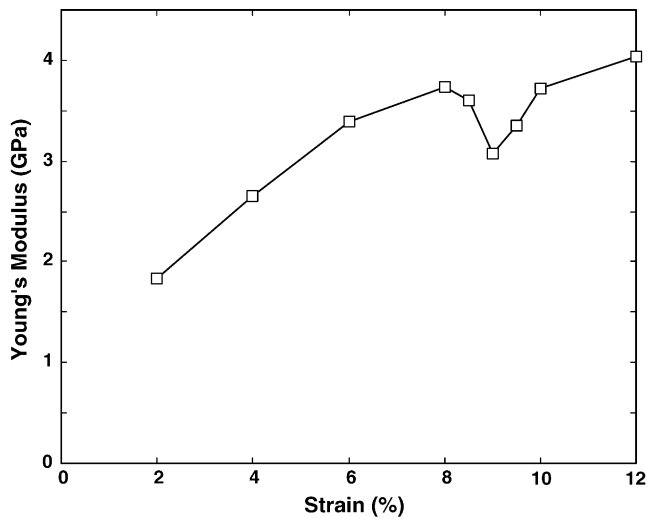
Fig. 11. Young's modulus vs. strain relationship of CuAlNi single crystal.

V-shape in the Young's modulus versus strain relationship is just a result of this, apart from similar pattern in the stress versus strain relationship. Since it is single crystal, the reorientation occurs in a relatively short strain range, i.e. abrupt in appearance. Consequently, the V-shape is rather limited within a small strain range.

The situation in polycrystals is a bit complicated because of the difference in grain orientation and constrain from neighbouring grains. However, the same mechanism found in the CuAlNi single crystal might be applicable for polycrystals as well. Assume that the individual grains follow the same behavior as that of the CuAlNi single crystal. Due to the interaction among grains, the occurrence of abrupt reorientation should become smoother over a far larger strain range. Therefore, the V-shape in the Young's modulus versus strain

relationship in polycrystals is in a more gradual manner. This is exactly what has happened in the tests.

5. Conclusions

The V-shape phenomenon in the Young's modulus versus strain relationship was observed in uniaxial tension on NiTi polycrystalline wires of different phases and uniaxial compression on an austenite CuAlNi single crystal. In all tests, the bottom of V-shape was found at round the point where hardening starts. Reorientation of martensite variant(s), which was clearly observed in CuAlNi single crystal by surface study, is suggested as the mechanism behind this phenomenon.

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