Structural-scale levels of development of inelastic martensitic deformation during isothermal loading of submicrocrystalline titanium nickelide in premartensitic condition

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Structural-Scale Levels of Development of Inelastic Martensitic Deformation during Isothermal Loading of Submicrocrystalline Titanium Nickelide in Premartensitic Condition

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Abstract. The results are presented of an experimental investigation into the regularities and mechanisms of the development of thermoelastic martensitic transformation in submicrocrystalline alloy $Ti_{49.4}Ni_{50.6}$ with different ways of thermo-power actions using the methods of optical microscopy in situ and X-ray diffraction. The peculiarities of localization of martensite transformation at the meso- and macroscale levels in this alloy with submicrocrystalline structure are considered. Experimental data on the relay mechanism of propagation of the martensitic transformation are presented. The interrelation between the localization of the martensitic transformation on the meso-and macroscale levels and deformation behavior under isothermal loading alloy $Ti_{49.4}Ni_{50.6}$ in submicrocrystalline condition are shown and discussed.

INTRODUCTION

With development of special methods of intensive plastic deformation the opportunity arose of obtaining alloys undergoing thermoelastic martensite transformations with an ultrafine-grained (nano- and submicrocrystalline) structure. One of them is TiNi alloy. Recent studies have shown that fragmentation of the grained structures to the submicrocrystalline state in Ti-Ni-based alloys caused a significant decrease in characteristic temperatures of martensitic transformations, an increase in reversible martensitic deformation, in reactive stresses and strength characteristics with the persistence of high plasticity.

It was previously found that alloys with a submicrocrystalline structure formed by severe plastic deformation have imperfect grain boundaries and small size of grains [1–8]. They contain new defects with uncompensated stress fields, which lead to an elastic deformation of the lattice and to the appearance of a long-range stress field at the boundaries and at triple junctions of grains. The nature of the involvement of individual grains and their complexes in martensitic transformation could be different than in alloys with the coarse-grained structure, because of the presence of stresses in alloys with submicrocrystalline structure at the thermal and mechanical actions.

RESULTS AND DISCUSSION

In this paper, some of the research results of development the thermoelastic martensitic transformation and their localization at the meso- and macroscale levels during isothermal loading in premartensitic condition of $Ti_{49.4}Ni_{50.6}$ submicrocrystalline alloy are presented. The results of similar studies on $Ti_{49.4}Ni_{50.6}$ alloy with a coarse-grained structure condition are shown and discussed in [3]. Studies have been carried out on a binary alloy $Ti_{49.4}Ni_{50.6}$ (%) with a small content by volume (~3 vol %) of particles of phase Ti_4Ni_2 (O, C, N). For the research flat samples were

used with two blades $2 \times 0.5 \times 5$ mm^3, which were cut out on an electric spark machine along the axis of cylindrical workpieces, and then were grinded and polished electrolytically.

The temperatures of martensitic transformations in the alloy are the following: the temperature of the beginning $T_R = 278$ K, $M_s = 257$ K, $A_s = 270$ K) and the end ($M_f = 242$ K, $A_f = 285$ K), the direct R(T_R) and B19′ (M_s, M_f) and reverse B19′ (A_s, A_f) transformations in the free state.

It was established that during isothermal stretching the alloy at $T_d = 298$ K (in the premartensitic state when $M_s < T_d < A_f$) inelastic martensitic deformation was accumulating in three stages, for each of them there are their own patterns of localization of martensitic transformation. Figure 1 shows a tension curve at 298 K of submicrocrystalline alloy Ti$_{49.4}$Ni$_{50.6}$, deformation rate was 10^{-4} s^{-1}.
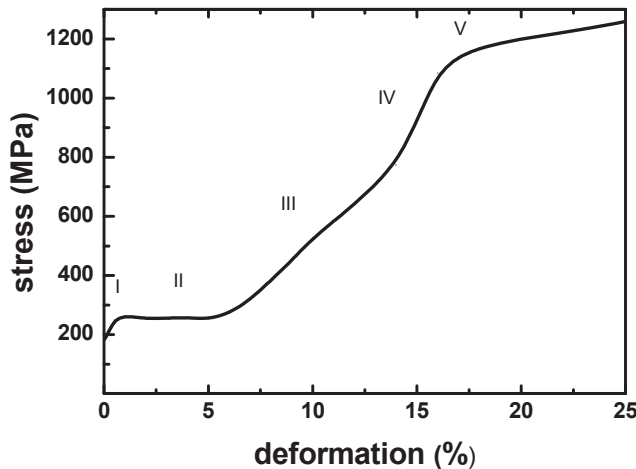
FIGURE 1. Tension curve at 298 K of submicrocrystalline alloy Ti$_{49.4}$Ni$_{50.6}$, deformation rate was $\dot{\varepsilon} = 10^{-4}$ s^{-1}

The observation *in situ* of the surface relief by the speed of stretching 10^{-4} s^{-1} showed that the start of the martensitic deformation in the first stage was connected with the formation of short mesobands of two orientations on one of the blades of the sample. They are presented as chains of the grains in the places where the inelastic martensitic deformation occurred (Figs. 2a and 2b)). They are oriented at an angle of approximately 60° to each other and to the axis of stretching of the sample. With an increasing of stress the quantity of mesobands and their length were increased (Fig. 2c). During the initial stage of mesobands growth when removed the applied stress mesobands were becoming shorter and disappearing entirely. This indicates the presence of large elastic stresses before the growing mesobands.

The growth of mesobands length with the increasing of the applied stress was occurring through the transfer of martensitic shift from grain to grain ("step by step"). A relay mechanism of involving grains in the martensitic transformation, as in the case of coarse-grained structure, was realized [3]. The growth of mesobands of localized martensitic transformation at an angle of about 60° to the stretching axis can be connected with the method of preparation of a submicrocrystalline structure, namely with equal channel angular pressing with an angle between the channels 110°. When the workpiece was passing through the channels maximal plastic shifts were occurring in the direction that divides the angle in half, i.e. an angle of approximately 55° to the axis of the workpiece. Namely in this direction mesobands were growing.

The end of the first stage (Fig. 1, I on the curve) of accumulation of inelastic martensitic deformation coincided with the arrival of mesobands to the opposite face of a sample (Fig. 2d). This was followed by the release of part of the accumulated elastic energy which led to an instantaneous appearance of a large amount of new mesobands of a localized martensitic transformation. Simultaneously the martensitic transformation was developing between created mesobands and was filling the intervals between them by formation of martensitic relief. Thus a macroband of localized martensitic deformation by the type band of Luders was formed and crossed the whole cross-section of the sample near the blade. The deformation rate 10^{-4} s^{-1} was low enough to avoid local overheating of the sample in the region of the front [4, 5].

The second stage on the stretching curve (Fig. 1, II on the curve) is associated with involvement in the martensitic transformation on the remaining part of the sample by means of a continuous motion of the macroband front of martensitic transformation along the sample while maintaining a constant value of the angle between the front of the macroband and the direction of stretching. Before promotion of front macrobands in the sample mesobands were growing continuously, whose length reached 250 μm with an average distance between them of about 40 μm. Completion of the front motion of martensitic transformation through the working part of the sample

coincided with the end of the second stage of the accumulation of martensitic deformation and it conforms to the end of playground yield.

X-ray studies showed that at the end of the playground yield the alloy was in a two-phase state: on the X-ray picture reflection of B19′ martensite and reflections of B2 phase have about the same intensity (Fig. 3, curve *2*). As we propose, the material at this stage is subjected to fragmentation with the formation of two types of zones: in the first type of zones martensite transformation is finished, and in other zones B2 phase is remained.

In the third stage of the inelastic martensitic deformation (Fig. 1, III on the curve) the surface relief gradually was becoming uniform and a conclusion can be made about the continuation of martensitic transformation. On the X-rays we can see that the intensity of the reflections B2 phase gradually decreases and the intensity of the reflections of martensite B19′ increases (Fig. 3, curve *3*).

Reverse martensitic transformation B19′ → B2 occurred after unloading at the end of the second stage and after subsequent heating. It was present as the motion of two fronts phase boundaries B2–B19' towards each another. Both fronts were moving simultaneously from the opposite faces of the sample, unlike the direct martensitic transformation when only one front was moving and other was fixed.
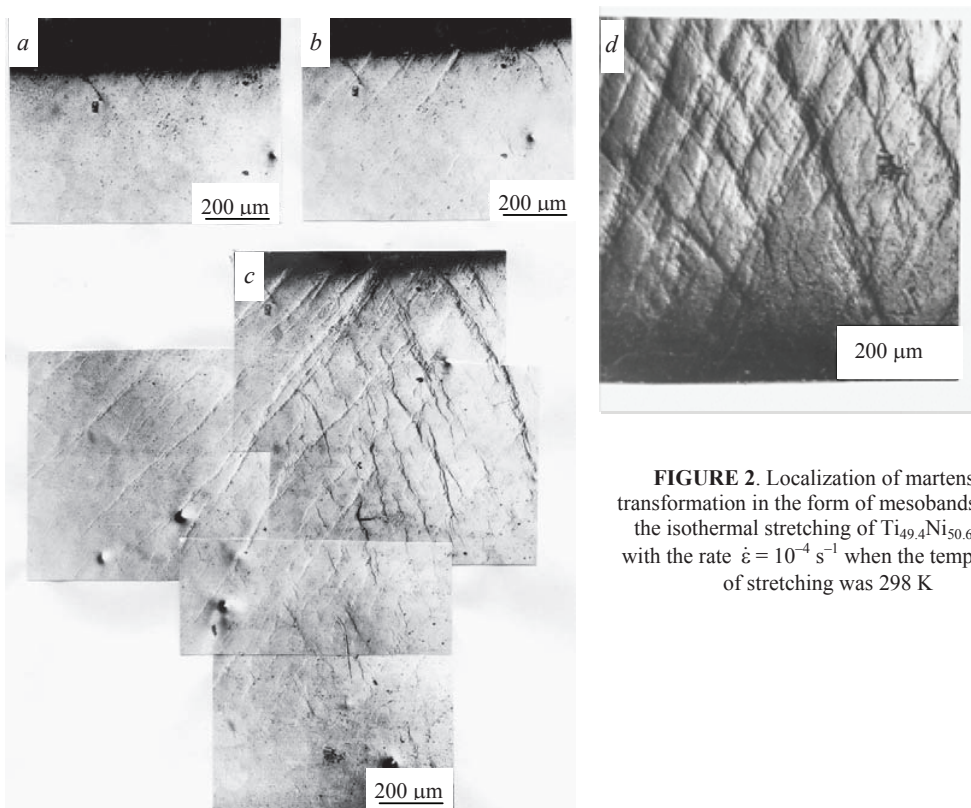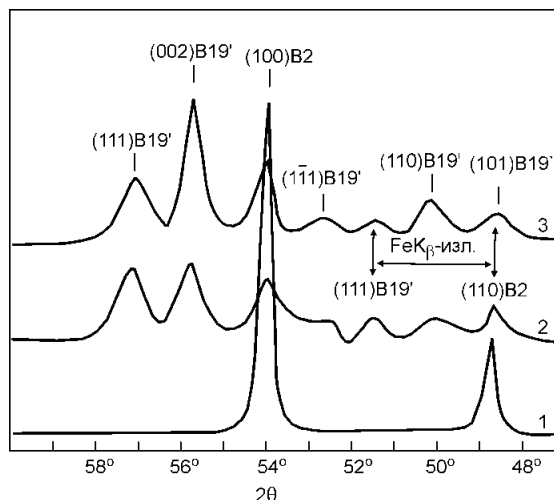

FIGURE 2. Localization of martensitic transformation in the form of mesobands during the isothermal stretching of $Ti_{49.4}Ni_{50.6}$ alloy with the rate $\dot{\varepsilon} = 10^{-4}$ s^{-1} when the temperature of stretching was 298 K

In some cases there was an interesting feature in the behavior of the fronts during heating of the sample after the second direct transformation and removal of the load. During the heating one of the fronts changed its orientation relative to the other and the axis of stretching on angle 60º. By the end of a reverse transformation we could see a macroband in the form of equilateral triangle, two sides of which were two fronts, which were located on the surface of the sample. Before this study we were unaware that there could be such an effect of the reorientation of the front during the time of the reverse martensitic transformation.

It should be noted that there are different ways of realization of inelastic strain on the second stage of the deformation curve. First, inelastic strain can be formed mainly due to the involvement of new areas of the material

during the martensitic transformation. Second, inelastic strain can occur through further development of transformations in the macroband. To investigate this question an experiment was performed.

Stretching stopped at different degrees of elongation of the sample; thereafter the length of that part of the sample, where the front of martensitic transformation passed, was determined by metallographic measurements. Assuming that this part of the sample became the cause of its elongation, we estimated the quantity of the local strain at different values of stretching of sample in this area. These measurements showed that the strain in the area where the macroband of martensitic transformation passed did not depend on the relative elongation of the sample. Its quantity was equal to the strain at the time of passage the macroband through the sample. Consequently, the inelastic martensitic deformation in the second stage of the curve was almost completely developed by involving new areas of material in a martensitic transformation by the type of Luders band.

FIGURE 3. X-rays of $Ti_{49.4}Ni_{50.6}$ alloy with submicrocrystalline structure in the initial state (1) in the II (2) and III (3) stages of deformation (K_β-radiation)

CONCLUSIONS

This work shows that deformation behavior of the submicrocrystalline $Ti_{49.4}Ni_{50.6}$ alloy under isothermal loading in a premartensitic condition is the same as in a coarse-grained structure.

The inelastic martensitic deformation is observed in three stages: the first stage is associated with the genesis, the second—with the spread of the macroband of martensitic transformation by the type of Luders band, the third—with additional transformation in the whole volume of the sample. However, the stresses of start and finish of every stage in the submicrocrystalline structure are 2 times higher at each following stage, than in the coarse-grained structure. The degree of deformation increases in all three stages, when the submicrocrystalline structure becomes coarse-grained.

The front of a macroband of martensitic transformation in an alloy with a submicrocrystalline structure forms an angle approximately 60º to the axis of the tension of sample. The development of martensitic transformation in mesobands begins earlier than the motion of the front of a macroband along the axis of the sample. The relay mechanism of involving the grains in martensitic transformation during isothermal loading is a consequence of the nonuniformity of the stress-strain state. Localization of inelastic martensitic deformation on meso- and macroscale levels is a consequence of the presence in alloy of meso- and macroconcentrators of stresses.

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