

DEFORMATION INDUCED MARTENSITE CHARACTERIZATION IN FE-30%NI-5%CU ALLOY

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Abstract

Deformation induced martensite properties were examined according to existing martensite morphology, crystallography and formation temperatures for different prior austenite homogenization conditions in Fe-30%Ni-5%Cu alloy. Scanning electron microscope (SEM), differential scanning calorimetry (DSC) and X-ray diffraction (XRD) techniques were employed to investigation. Scanning electron microscope observations showed elongated deformation induced martensite morphology in the austenite phase of alloy. As well, after deformation martensite start temperatures (M_s) were determined as -101°C and -105°C from DSC measurements for different homogenization conditions. In addition, X-ray diffraction analysis revealed the face centred cubic (fcc) of austenite phases and body centred cubic (bcc) deformation induced martensite phases for all studied samples.

Keywords: FeNiCu; SEM; DSC; XRD; Phase transformations; Martensite.

1. Introduction

Martensite, the product phase of a martensitic phase transformation in many steels and ferrous alloys have been attracted

too much interest in past few decades because of its desirable technological features in industry and technology [1-5]. Anyway, this unique microconstituent forms by applying various external driving forces

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such as thermal affect (cooling), deformation affect, external magnetic field and hydrostatic pressure to the austenite (gg) phase of studied ferrous alloys [6-9].

Noteworthy, these various external forces for inducing a martensitic transformation in the austenite phase of related specimen alter some transformation characteristics especially transformation kinetics, existing martensite morphology and crystallography [10, 11]. As well pointed out by Durlu [12] in his previous report, deformation induced martensite in ferrous alloys forms in needle like shapes but butterfly (or chevron) like morphologies and contains high densities of dislocations when compared with thermally induced martensite. Additionally, in general, thermally induced martensite shows bcc crystal lattice (a' martensite) at a M_s formation temperature. On the other hand, deformation induced martensite shows a crystallographic variety from bcc to hexagonal closed packed (hcp) crystal structures (martensite) at a M_d formation temperature [13, 14]. However, several ferrous alloys display more than one martensite crystal type depending on the alloy composition. In the some ranges of composition both crystal structures may coexist, for example a' by deformation affect and e martensites by thermal affect in Fe-Mn alloys and deformed metastable i.e, 301, 302, 304, 304L, 316 and 316L steels [15, 16]. Besides, these mentioned diversities of martensite formation in ferrous alloys leads researchers to a continuous interest for clarifying and understanding the martensite

formation under various distinct conditions

Overall, martensite formed by deformation may be different from that formed by cooling according to formation nature, existing martensite morphology, formation temperature and crystallography and is still focus of recent investigations. Therefore, the aim of the present study was to clarify the some curious characteristics of the deformation induced martensite in Fe-30%Ni-5%Cu alloy according to existing martensite morphology, formation temperatures and crystallography of both austenite and martensite phases.

2. Experimental

2.1 Alloy and Heat Treatments

The alloy used in the present study was Fe-30%Ni-5%Cu (wt %) which was prepared by vacuum induction melting under an argon atmosphere from pure (99.9 %) alloying elements. After this alloying, product alloys were cylindrical bars with 1cm diameter and 10 cm length.

To examine the quenching media affect on deformation induced martensitic transformations, we designated two heat treatments. Firstly, group A samples were homogenized at 1000 °C for 24 hours and then cooled in furnace to room temperature. Likewise, group B samples were homogenized at 1000 °C for 24 hours and quenched into water at room temperature. Besides, A1 and B1 demonstrate the pure austenite phases for group A and group B, respectively (Table 1).

Table 1. Experimental details of studied Fe-30%Ni-5%Cu alloy.

Sample	Homogenization	Microstructure	M_d Temperatures (°C)
A1	1000°C→24h→Furnace cooling	Austenite	-
A2	-	Martensite	-101
B1	1000°C→24h→Water quenching	Austenite	-
B2	-	Martensite	-105

2.2 Compression Deformation Tests

Cubic compression samples of dimensions 4 x 8 x 4 mm were mechanically cut from the austenitized samples of both groups A and B. Compression test machine was an Instron 8510. All compression deformation tests were performed at room temperature with a 0.40 mm/min cross-head speed. Two samples from group A and group B deformed 40% with the same deformation amount. These deformed samples were denoted as A2 and B2, respectively.

2.3 SEM Observations

Details of microstructures were performed with a JEOL-JSM-5600 type scanning electron microscope during this research. After heat treatments and compression deformation tests, sample A1, A2, B1 and B2 were cut mechanically ~ 150 μm thickness and mechanically polished with diamond pastes through a conventional manner. All samples for microstructural observations were etched in a chemical solution 5ml HF+20 ml H₂O₂+ 25ml H₂O. Prepared samples then were examined in SEM with 20 kV operating voltage.

2.4 DSC Measurements

Differential scanning calorimeter technique was used during study to find out and compare M_d temperatures after deformation. New samples were prepared from A2 and B2 in the shape of 3mm radius discs and encapsulated in aluminium pans. Differential scanning calorimeter measurements of these samples were taken

by using a Perkin-Elmer Sapphire model thermal analyser. Cooling rate of the samples was measured by 10 °C/min. The studied temperature range was between 25°C and -150 °C during DSC measurements.

2.5 XRD Measurements

To clarify the crystal structures of deformation induced martensite phases, X-diffraction technique was employed to study. For XRD measurements, powder samples from B1 and B2 were prepared mechanically. After this preparation, powder samples were measured at room temperature in a Rigaku Geigerflex D-MaxB X-ray diffractometer with Cu-K α radiation source and monochromator.

3. Results and Discussion

3.1 SEM Observations

Fig. 1a and Fig. 1b shows the austenite phases of sample A1 and B1 with typical austenite grain boundaries, respectively. Also, Fig. 2a and Fig. 2b show the SEM micrograph of sample A2 and sample B2 taken after 40% deformation at room temperature. As can be clearly seen from Fig. 2a and Fig. 2b deformation induced martensite displays some more extended martensite crystals because of deformation affect and amount when compared with previously observed thermally induced martensite crystals for Fe-Ni-X alloys [17-19].

From a morphological viewpoint, deformation induced martensite in samples A2 and B2 displays extended and lengthened shapes as in typical deformation induced

martensite in ferrous alloys [20-22].

3.2 DSC Measurements

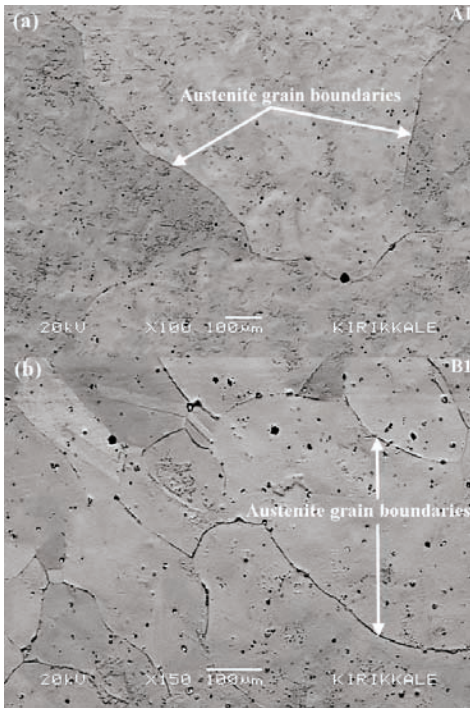


Fig. 1 SEM images of samples A1 and B1

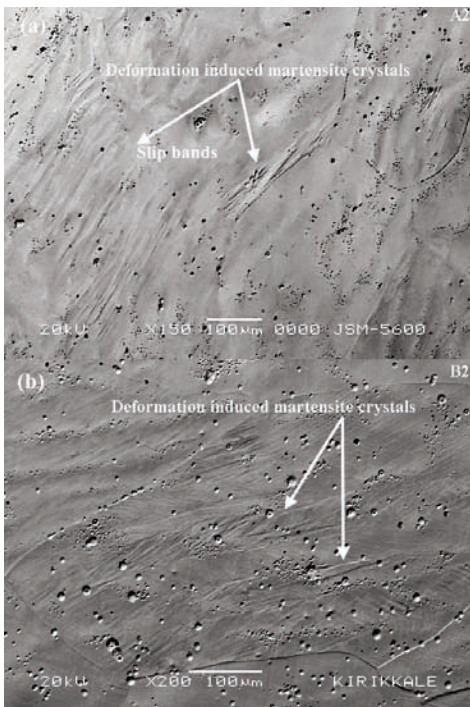


Fig. 2 SEM images of samples A2 and B2.

Fig. 3a shows a DSC curve of sample A2. According to this curve, M_d temperature of sample A2 appears at $-101\text{ }^\circ\text{C}$. However, M_d temperature of sample B2 appears at $-105\text{ }^\circ\text{C}$ as seen from below DSC curve in Fig. 3b. Although same amount of deformation was applied on both samples A2 and B2, martensite formation temperatures display a small difference. This main difference for M_d temperatures between sample A2 and B2 originates absolutely from the prior austenization conditions. Present results for this case obviously supports Nishiyamas [13] detailed evaluation. According to his explanation, in the same alloy, prior austenite homogenization conditions can change some transformation characteristics such as existing martensite kinetics, morphology, and formation temperatures. As a result,

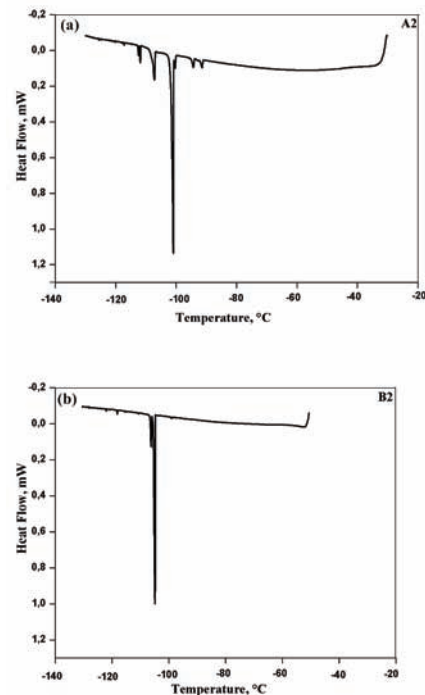


Fig. 3 DSC curves of samples A2 and B2.

changing of quenching media during austenization changed the deformation induced martensite formation temperatures. Anyway, this result associates with the formation of prior austenite phase during austenite-martensite phase transformations. That is, slow cooling (cooling in furnace) causes larger austenite grains with respect to fast cooling (water quenching). Finally, dislocation motion in a larger grained sample may be easier relative to a smaller grained sample since smaller grained austenite phase has a greater total grain boundary area which impedes the motion of dislocations. In other words, martensite formation in a larger

grained sample becomes easier than the martensite formation in a smaller grained austenite [20].

3.3 XRD Measurements

Fig. 4 shows X-ray diffraction patterns of samples A2 and B2 respectively. These X-ray diffraction patterns reflect the crystal characters of martensite phases with their crystallographic planes. Moreover, deformation induced bcc martensite (α') peak intensity arises in sample B2 when compared with sample A2. The distinguished X-ray diffraction patterns in samples A2 and B2 mainly originates from the abovementioned prior austenite homogenization conditions and grain sizes of both samples.

4. Conclusions

i. According to present experimental results, deformation induced martensite in the austenite phase of Fe-30%Ni-5%Cu alloy appears with extended and lengthened shapes after 40 % deformation as in many ferrous alloys (Fig.2a and Fig. 2b). These martensite crystals display a distinguished morphologic character when compared with recently published [23] thermally induced thin plate martensites in Fe-30%Ni-5%Cu alloy.

ii. Different quenching media at constant homogenization temperature (1000°C) obviously alter the M_d temperatures (Fig. 3a and Fig. 3b). Further, M_d temperatures after deformation were obtained as -101°C and -105°C which are higher than $M_s = -122^{\circ}\text{C}$ [23] under similar heat treatment in the same

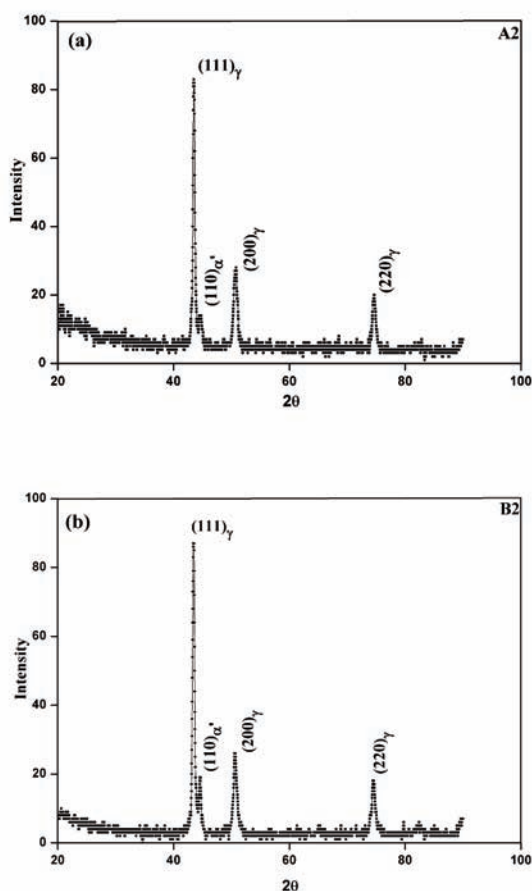


Fig. 4 XRD patterns of samples A2 and B2

Fe-30%Ni-5%Cu alloy. This finding presents a fair agreement with the well-known information that M_d temperature after deformation of a ferrous alloy can be higher than thermally induced (cooling affect) M_d temperature of same alloy [13].

iii. From a crystallographic aspect, deformation induced martensite shows a bcc crystal structure (Fig.4) as in thermally induced martensite [23] within prior austenite phase of the Fe-30%Ni-5%Cu alloy under studied conditions.

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