Fects of the Pre-Strain on the Kinetics and Volume Fraction of Martensitic Phase Transformations in an Fe-30% Ni-3% Pd Alloy

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Abstract: In this study, kinetics, morphological and magnetic properties of thermal induced and pre-strain of austenite martensitic transformations in an Fe-30%Ni-3%Pd alloy were investigated. Effect of pre-strain to austenite phase above the $M_s$ temperature and kinetics, morphological and magnetic properties of martensitic transformations which was formed by below the $M_s$ temperature after the pre-strain were investigated. Kinetics of martensitic transformation (martensite start temperature ($M_s$), austenite start temperature ($A_s$) and austenite finish temperature ($A_f$) were determined by Differential Scanning Calorimetry (DSC). Kinetics of the transformation was found to be as athermal type and $M_s$ temperature decreased with increasing prestrain. 5% and 10% pre-strain which was applied to austenite phase at room temperature formed slip lines. Martensite plates, which were broken and bent, formed by cooling the alloy after 5% prestrain and martensite plates which were decrescent formed by cooling after 10% pre-strain, observed by Scanning Electron Microscopy (SEM). The volume fraction of martensite and austenite phases, the hyperfine magnetic field of martensite phase and isomery shift values in Fe-30%Ni-3%Pd alloy have been determined by using Mössbauer Spectroscopy. The pre-strain which are influential on the transformation kinetics and the martensite plates morphology, increased the rate of martensite volume.

Key words: Fe Ni Pd Alloy • Martensitic Transformations • Electron Microscopy • Mössbauer Spectroscopy • Pre-Strain

INTRODUCTION

The martensitic transformation is one of typical first-order structural phase transitions without atom diffusion and has been widely studied to determine its characteristics from physical, metallurgical and crystallographic view points [1]. Early studies on the formation of martensite in different ferrous alloys revealed that the formation mechanism and substructure of martensite are considerably altered by the transformation temperature, alloy composition, strengths of the material in the austenite and martensite phases, cooling rate of the matrix structure during the transformation and the austenite stacking fault energy in addition to the well known effects of the austenite grain size and the formation sequence of the martensitic structures [2-5].

A significant property of martensitic phase transformations in Fe-Ni alloys, the morphology of martensite dependent on Ni percentage [6]. Furthermore, addition of Mn, Co, C, etc. as a third alloying element to binary Fe-Ni alloys changes some properties of martensitic transformations such as kinetics of the transformation and morphology of the martensite [7-9]. Martensitic transformation on cooling begins at the $M_s$ temperature; the extent of the transformation progressively increases with lowering of temperature and it is completed, finally attaining the complete transformation at the temperature $M_f$, which is known as the martensite finish temperature [10]. In many of the martensitic transformations discussed so far, the reactions start at the $M_s$ temperature and proceed while the temperature is falling. When the cooling is stopped, the reaction stop and when the cooling is resumed, they start...
again. The reactions proceed only while the temperature is changing [1]. Therefore, martensite produced by this type of reaction is referred to as athermal martensite.

From a magnetic viewpoint of Fe-based alloys, another significant property of the martensite is its distinguished magnetic character from austenite. In spite of the paramagnetic nature of the austenitic parent phase in these alloys, martensitic product phase can show either ferromagnetic or antiferromagnetic behaviours [11].

Although many studies are present for thermally induced martensite in various Fe-Ni-X alloys, no work has reported for the Fe-30%Ni-3%Pd alloy. Therefore, the present study was to examine, morphology and martensite kinetics of thermally induced martensite in Fe-%30 Ni-%3Pd alloy by SEM, DSC and Mössbauer spectroscopy.

**Experimental:** The alloy employed in the present study was Fe-30%Ni-3%Pd (wt.%) which was prepared by vacuum induction melting under an argon atmosphere from pure (%99.9) alloying elements. This alloy was cylindrical bars (1cm diameter and 10 cm long). Bulk samples were homogenized at 1100°C in quartz tube for 6 h and then quenched in water at room temperature. These samples then mechanically thinned for SEM pre-strain. Scheil has presented metallographic evidence to show that this effect is due to the slip bands acting as barriers in the same way as grain boundaries and limiting the size of the martensite plates that can form [13].

According to the SEM observations, the size of martensite plates decreased with the increasing rate of pre-strain. Scheil has presented metallographic evidence to show that this effect is due to the slip bands acting as barriers in the same way as grain boundaries and limiting the size of the martensite plates that can form [13]. Therefore, martensite plates in iron-nickel alloys are seen to pass through slip bands and even through deformation bands. Besides, at greater levels of austenite pre-strain, the nature of the transformation at the burst remains autocatalytic, but the martensite plate size continually decreases and the arrays of plates follow selected growth paths leaving large areas of austenite untransformed [14].

**RESULTS AND DISCUSSION**

Information of the microstructure of plastically deformed specimens was obtained from SEM observation. Figure 1 illustrates SEM image of deformed samples by 5% and 10% pre-strain. The surface of a metal crystal which has been polished, then plastically deformed, becomes covered with one or more sets of parallel fine lines called slip lines [12]. Figure 1 shows typical slip bands on the surface of an Fe-30%Ni-3%Pd alloy deformed at room temperature.

Figure 2 shows a SEM micrographs of this samples after the 5 s liquid nitrogen (-196°C) immersion. This thermal stimulation caused martensite plates formation in figure 2.

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![Fig. 1: SEM micrograph of (a) % 5 and (b) %10 pre-strained samples of Fe-30%Ni-3%Pd alloy.](image-url)
In order to determine the effects of the pre-strain on the martensitic transformations kinetics in the Fe-30% Ni-3%Pd alloy have been studied. Martensite start temperature ($M_s$) of martensite transformation was determined by differential scanning calorimetry (DSC) measurements as -33°C and -40°C respectively (fig. 3). Some evidences such as new nucleation sites or growth of existed martensite crystals achieved by further isothermal martensitic transformations are well described by Kajiwara [15]. Although sample of the Fe-30% Ni-3% Pd alloy were kept at -196°C for 10 min, 10 h and 10 days, these isothermal holding times didn’t cause the formation of martensite crystals. This application suggest that the martensite kinetics displayed athermal type.

According to the DSC results, the $M_s$ temperature decreases as the level of pre-strain. Such a depression of $M_s$ temperature as a function of pre-strain is characteristic of a wide variety of ferrous alloys. The generally accepted view is that strain hardening of the austenite prior to transformation increases the resistance of the lattice to the shear transformation and thus decreases the $M_s$ temperature [14]. Therefore, the $M_s$ temperature is depressed as a function of pre-strain.

Mössbauer spectra observed for the specimens of the examined alloy at room temperature is shown in figure 4. A typical six-line spectrum of the ferromagnetic structure and also a singlet corresponding to the matrix austenite were observed after the heat treatments as reported earlier [11]. The Mössbauer parameters such as isomery shift and hyperfine magnetic field are given in Table 1 with the calculated volume fractions of the each phase. According to the Mössbauer results, the volume fraction of martensite phase increases as a function of pre-strain level.
Fig. 4: Mössbauer spectra of (a) 5% and (b) 10% pre-strained samples of Fe-30%Ni-3%Pd alloy, single peak represents the retained paramagnetic austenite phase and sextet represents the ferromagnetic athermal martensite phase.

Table 1: Some Mössbauer parameters of 5% and 10% pre-strained samples of Fe-30%Ni-3%Pd alloy.

<table>
<thead>
<tr>
<th>Percentage of pre-strain</th>
<th>A(%)</th>
<th>M(%)</th>
<th>$\delta_\alpha$</th>
<th>$\delta_m$</th>
<th>$H$ (T)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>16.72</td>
<td>83.28</td>
<td>0.50577</td>
<td>0.47641</td>
<td>32.976</td>
</tr>
<tr>
<td>10</td>
<td>10.49</td>
<td>89.51</td>
<td>0.13325</td>
<td>0.35641</td>
<td>32.073</td>
</tr>
</tbody>
</table>

**CONCLUSIONS**

- According to DSC results, the martensitic transformations kinetics found as athermal, namely martensitic phase transformation displayed the burst phenomenon in the studied alloy. Also, the $M_s$ temperature decreases and depressed as a function of pre-strain.
- 5% and 10% pre-strain which was applied to austenite phase at room temperature formed slip lines.
- From a morphologic viewpoint, thermally induced martensite exhibits plate morphology and the size of martensite plates decreased with the increasing rate of pre-strain.
- Martensite plates, which were broken and bent, formed by cooling the alloy after 5% pre-strain and martensite plates which were decrescent formed by cooling after 10% pre-strain, observed by Scanning Electron Microscopy (SEM).
- Mössbauer studies shows that, the volume fraction of martensite phase increases as a function of pre-strain.

**REFERENCES**