Effects of Specimen Thickness on the Temperature of Nucleation of Martensite in Fe-Ni Alloys

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$M_s$ ($M_b$) temperatures of thin Fe-Ni foils were measured as a function of thickness. It is found that two opposing effects yield a scatter of $M_s$ ($M_b$) to lower as well as to higher temperatures. These effects can be related to the distribution of nuclei per unit volume and to the effects of image forces on martensite nuclei in thin foils.

The upper temperature limit of the formation of martensite, $M_s$, has been observed to increase if the specimen thickness, $t$, is reduced below about 1 µm (1-3). However, measurements of the function $M_s(t)$ have not been reported. Since the structure of the nucleus and the mechanism of nucleation of martensite are still unknown (e.g. 4) any measurement and structural observation related to the nucleation process may contribute to its final analysis. Therefore, the present measurements of $M_s(t)$ of 4 Fe-Ni alloys were performed and the results are discussed with respect to their relevance to the nucleation problem.

EXPERIMENTAL PROCEDURE

The Fe-Ni alloys whose chemical analyses are given in Table I were prepared in two ways. Alloys 1 to 3 were made from carbonyl-Ni (C quality) and carbonyl-Fe (CCS quality) by powder metallurgy techniques involving sintering for 8 hr. at 1400°C. The average diameter of the powder particles was <10 µm such that the sintering treatment was, also, sufficient for complete homogenization. The alloys were then cold rolled to strip of ≤3 µm thickness with several intermediate anneals.

Alloy 4 was induction melted from Ni (99.99) and Fe (Ferrovac E) to an ingot which was subsequently cold rolled to strip of ≤15 µm thickness with intermediate anneals. The strips were cut into squares of 5 by 5 mm. All alloys were then austenitized under vacuum for the times and at the temperatures indicated in Figs. 3(a) to 3(d).

In order to obtain specimens of different thicknesses, the blanks were placed on a flat surface of an insulating and acid resistant material and electropolished by directing a stream of electrolyte onto the exposed surface, see Fig. 1. The electrolyte consisted of chromium trioxide, acetic acid, and water as commonly used in preparing thin foils for electron microscopy. The thinning rate was first determined by trial and error and was then used for timing the thinning runs to obtain different thicknesses in the bottom of the resulting shallow dimple.

The measurements of $M_s$ which followed next were based upon microscopic observation (X36 magnification, 3 mm diam field of view) of the surface relief produced by the first martensite plates to form. The specimen was placed on the plane and polished end of a cylindrical copper slug standing in a container into which some liquid nitrogen was filled such that the specimen was cooled down slowly by conduction through the copper slug, see Fig. 2. The temperature was measured using a copper-constantan thermocouple clamped to the surface next to the specimen and the temperature at which the first martensite plate became visible was recorded.

The thickness was measured by X-ray absorption. To this end, the specimens were attached with vaseline to a holder placed between the X-ray tube and a counter. A beam of 0.12 mm diam was directed normal to the plane of the specimen. The holder permitted displacement of the specimen in two directions normal to the beam such that the thinnest area could be selected for

Table I. Alloy Compositions

<table>
<thead>
<tr>
<th>Alloy No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt. pct Ni</td>
<td>30.0</td>
<td>30.9</td>
<td>32.1</td>
<td>32.6</td>
</tr>
</tbody>
</table>

Fig. 1—Set-up for electrolytic thinning.
measurement by adjusting for the highest counting rate. The measurements were evaluated based upon calibration with specimens whose thickness had been determined mechanically. The overall accuracy of the average thickness of the irradiated specimen area obtained from these measurements is about ±10 pct.

The specimens were transferred with extreme care from the quartz capsule in which they had been austenitized to the thinning set-up, and copper slug for \( M_s \) determinations. Thus, possible effects due to elastic stresses and plastic deformation by bending and surface damage were minimized.

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**RESULTS**

All observations of \( M_s \) temperature, location of the first martensite plate formed in the specimen and effect of a reannealing treatment are shown in Figs. 3(a) to 3(d). It was not possible to distinguish clearly in all cases between the formation of individual plates (\( \geq M_s \)) and of groups of plates in a burst (\( \geq M_b \)). Therefore, in referring to the experimental results \( M_s \) will be used throughout the paper. It should be noted that the results from alloys 1 to 3 and those from alloy 4 are characterized by two essential experimental differences: the initial specimen thickness and most of the reduced thickness values for which \( M_s \) was determined were considerably lower for alloys no. 1 to 3 than for alloy no. 4. The findings can be summed up as follows:

1) above about 2 \( \mu \)m thickness, the scatter of the \( M_s \) temperatures decreases to a comparatively small range with increasing nickel content: >150°C for 30.0 wt pct Ni, 60°C for 30.9 wt pct Ni, 30°C for 32.6 wt pct Ni;

2) below about 2 \( \mu \)m thickness, the \( M_s \) temperatures assume a wider spread in the thinnest areas of the specimens: >220°C for 30.0 wt pct Ni, 160°C for 30.9 wt pct Ni, >120°C for 32.1 wt pct Ni; it is surmised that in alloy 4 the number of \( M_s \) measurements at small values of \( t \) was too small to show a similar spread as for alloys 1 to 3;

3) \( M_s \) temperatures above the range of bulk values are only found in the thinned portion of the specimens whereas if nucleation takes place first near the rim,

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![Fig. 2—Set-up for \( M_s \) measurements.](image)

![Fig. 3—(a) to 3(d). Results of \( M_s (t) \) measurements. Legend for 3(a) to 3(c): ● location of first plate unnoted; ▲ location of first plate unnoted, transformation above room temperature; A location of first plate near center, transformation above room temperature; ○ location of first plate near center; ○ location of first plate near rim; ◇ complete transformation by burst; ▼ transformation below −186°C; ◇ \( M_s \) of bulk material according to Ref. 5. Legend for 3(d): ● first measurement; ■ measurement after reannealing 30 min, 900°C; ◇ \( M_s \) of bulk material according to Ref. 5.](image)