Effect of extrinsic grain-boundary dislocations on M$_{23}$C$_6$ precipitate nucleation in an austenitic stainless steel

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From a comparison of the isothermal precipitation curve for M$_{23}$C$_6$ grain-boundary precipitates in an austenitic stainless steel and of the spreading times of extrinsic grain-boundary dislocations (EGBDs), it has been shown that M$_{23}$C$_6$ precipitates cannot directly nucleate on EGBD lines lying on random high-angle grain boundaries. This process can occur only on coherent and incoherent twin and other special boundaries.

1. Introduction
The grain-boundary precipitation process is the determinant of a number of properties of aged alloys. It seems that one of the important factors connected with grain-boundary precipitation is the dependence of the precipitate nucleation process on grain-boundary dislocations.

Recently Jones et al. [1-3] investigated precipitation of NbC particles at grain boundaries in an austenitic stainless steel. They concluded that extrinsic grain-boundary dislocations (EGBDs) were the principal nucleation sites. Pumphrey and Gleiter [4], however, have found that EGBDs in austenitic steel are incorporated in a boundary by a spreading of their cores after 30 sec heating at 500°C. Alternatively, Pond and Smith [5] suggested that incorporation of EGBDs into the grain boundary occurs by dissociation of these dislocations followed by line rotation. However, other recent experimental [6] and theoretical [7, 8] work supports the idea that the spreading process occurs by the widening of the EGBD core.

As the precipitation temperature of NbC particles was 930°C, the question arises as to how NbC particles could nucleate at this temperature on the EGBD lines which disappeared at approximately 500°C.

To explain the role of EGBDs in precipitate nucleation, the times required for EGBDs spreading at constant temperature were compared with the isothermal precipitation curve for M$_{23}$C$_6$ carbides at grain boundaries determined with the aid of an electron microscope.

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Si</th>
<th>Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.142</td>
<td>22.45</td>
<td>19.08</td>
<td>0.56</td>
<td>1.20</td>
</tr>
</tbody>
</table>

2. Experimental details
The material used in the investigation was an austenitic stainless steel of the composition shown in Table I. The initial phase which precipitates at grain boundaries in this particular type of steel during the heat-treatment procedure used is M$_{23}$C$_6$ carbide [9].

Rods, 3 mm diameter, were solution-treated at 1150°C for 1 h and subsequently quenched in water. Thin foils of the above material were observed in a Philips EM 300 electron microscope. At grain boundaries, no small precipitates were observed; large M$_{23}$C$_6$ carbides were occasionally seen both at grain boundaries and in the matrix. It can, therefore, be concluded that the cooling rate was sufficient for a solid solution to be obtained.

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Solution-treated rods were cut into slices, about 0.2 mm thick, to be aged in the temperature range 500 to 1000 °C, for different periods of time and then again quenched in water. Grain boundaries in such foils prepared from the above material were subsequently examined to obtain an isothermal precipitation curve for M23C6 grain-boundary precipitates.

Several rods were slightly deformed in the supersaturated state and after being aged for 50 sec at 700 °C. Thin foils were then examined in the heating holder of the electron microscope in order to observe the spreading of extrinsic grain-boundary dislocations. Observation of annealed boundaries was made with a change in the excitation of the first condenser lenses and with the excitation of the second condenser lenses adapted to the former. The illuminated area on the specimen is then considerably smaller. This observation technique developed from the fact that the heating sequence with an electron beam switched off, which was used by Pumphrey and Gleiter [4], did not show any positive results because of the very rapid oxidation of the thin foil.

### 3. Correlation between the nucleation of M23C6 carbides at grain boundaries and the spreading of EGBDs

The times elapsed before the first carbides were observed on grain boundaries during ageing in the temperature range 500 to 1000 °C are shown in Table II. After ageing, during periods of time shorter than those presented in Table II, no precipitates at any grain boundary were observed (about one hundred boundaries were examined for each particular temperature). Typical examples of images of precipitated carbides are shown in Fig. 1. On the basis of the data given in Table II, an approximate isothermal precipitation curve for M23C6 grain-boundary precipitates was drawn (Fig. 2). The disappearing times of EGBDs are also marked. Spreading experiments were made on grain boundaries with precipitated carbides (after ageing at 700 °C for 50 sec deformation), and also on precipitate-free boundaries (after solution-treatment and deformation). Detailed

<table>
<thead>
<tr>
<th>Ageing temperature (°C)</th>
<th>Time (sec)</th>
<th>Ageing temperature (°C)</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>7800</td>
<td>800</td>
<td>5–10</td>
</tr>
<tr>
<td>600</td>
<td>1800</td>
<td>900</td>
<td>2–5</td>
</tr>
<tr>
<td>700</td>
<td>30–40</td>
<td>1000</td>
<td>1–5</td>
</tr>
</tbody>
</table>

**Figure 1** Early stages of M23C6 precipitation at grain boundaries after ageing: (a) at 500 °C for 7800 sec; (b) at 600 °C for 1800 sec; (c) at 700 °C for 30 sec; (d) at 900 °C for 5 sec.