Formation of lamellar $M_{23}C_6$ on and near twin boundaries in austenitic stainless steels

B SASMAL

Department of Metallurgical Engineering, Indian Institute of Technology, Kharagpur 721 302, India

Abstract. Thin foil electron microscopy studies were made on the precipitation of lamellar $M_{23}C_6$ during aging at 973 K and 1073 K in water-quenched specimens of two austenitic stainless steels. After the precipitation on incoherent twin boundaries $M_{23}C_6$ formed on coherent twin boundaries and in the regions adjacent to incoherent twin boundaries. These precipitates showed lamellar morphology and were aligned in a specific manner with respect to the twin boundaries. Such lamellar precipitates were absent in the specimens which were isothermally treated at 1073 K after being transferred from the solution treatment temperature. The lamellar morphology of $M_{23}C_6$ is suggested to be developed by the influence of residual specific stress field around twin boundaries resulted from quenching.

Keywords. Electron diffraction; lamellar $M_{23}C_6$; precipitation morphology; twin boundaries.

1. Introduction

Precipitation of $M_{23}C_6$ in austenitic stainless steels was studied by many investigators. In quenched and aged conditions $M_{23}C_6$ precipitates (Lewis and Hattersley 1965; Wolff 1966; Beckitt and Clark 1967; Singhal and Martin 1967; Wilson 1971; Weiss and Stickler 1972) on incoherent twin boundaries after the precipitation on grain boundaries. During the later stages of aging, precipitation occurs on coherent twin boundaries and within the grains. The precipitates adjacent to an incoherent twin boundary grow as plates on both sides of the boundary, which are parallel to the coherent twin boundaries (Lewis and Hattersley 1965; Beckitt and Clark 1967; Singhal and Martin 1967; Hattersley 1964 and Lewis 1964).

Two models have been put forward to explain the formation of these lamellar precipitates. Beckitt and Clark (1967) suggested that the stress generated due to growth of precipitates nucleated on an incoherent twin boundary drives the twinning dislocations out of the boundary. These dislocations have been attributed to act as sites for nucleation continuously as they move out into the matrix and thus to form the plates of $M_{23}C_6$ on the $\{111\}$ (twinning) planes. Singhal and Martin (1967) however observed the stacking faults formed possibly by bowing of the twinning dislocation out of the incoherent twin boundary after the precipitation on the boundary. They have suggested that $M_{23}C_6$ precipitation occurs by nucleation along the bounding Shockley dislocation and by growth across the stacking fault to form a sheet of carbide. Shockley partial might again be repelled by the precipitate and the repetition of the precipitation process might cause further growth of the plate.

In order to examine these models a detailed electron microscopy study was made on

the formation of lamellar $M_{23}C_6$ adjacent to incoherent twin boundaries and on the precipitation at coherent twin boundaries.

2. Experimental

The chemical composition of two austenitic stainless steels (AISI 316L and DIN 4981, hereafter referred to A and B respectively) studied are given in table 1. The cold-rolled sheet specimens (about 150 μm thick) were solution-treated (5.4 Ksec (1 hr 30 min) at 1433 K for steel A and 1.8 Ksec (30 min) at 1550 K for steel B) inside sealed silica tubes in argon atmosphere and quenched in water. The quenched specimens were then aged at 973 K and 1073 K inside evacuated silica tubes for various durations from 3.6 Ksec (1 hr) to 14.4 Msec (4000 hr). A few bulk specimens (10 $\times$ 5 $\times$ 3 mm) of A after being solution-treated were transferred to 1073 K, held for 360 Ksec and 1.8 Msec and then cooled in air. Thin foils for electron microscopy examination were prepared by twin-jet electrolytic polishing with a mixture of 10% perchloric acid and 90% acetic acid.

3. Results and discussion

Steel A was fully austenitic in the as-quenched condition, while B contained a considerable amount of undissolved NbC particles. $M_{23}C_6$ was the only carbide to be precipitated in A during aging at 973 K and 1073 K. In B both NbC and $M_{23}C_6$ formed. Because of the precipitation of NbC in very fine form on grain boundaries, on dislocations, in association with stacking faults and as dot-like particles, the precipitation of $M_{23}C_6$, in general, was appreciably low and was delayed in B (Sasmal 1980). $M_{23}C_6$ first precipitated on grain boundaries and later on incoherent twin boundaries followed by intragranular precipitation on continued aging. After the precipitation on incoherent twin boundaries a preferential precipitation of $M_{23}C_6$ was noted in the regions adjacent to these boundaries (figure 1). Precipitation on coherent twin boundaries also occurred simultaneously. In B, though the intragranular precipitation was rarely observed, a new mode of precipitation of $M_{23}C_6$ around undissolved NbC particles was noted. This is reported earlier (Terao and Sasmal 1978, 1981).

Electron diffraction analysis showed that the $M_{23}C_6$ precipitates were face-centred cubic and had almost the same lattice parameter in both the steels: $a = 10.7 \text{ \AA}$. They were found to grow at all the sites with the cube orientation relationship with austenite matrix, i.e.

$$\begin{aligned} \{100\}_{M_{23}C_6} &\parallel \{100\}_\gamma, \\ \langle 100 \rangle_{M_{23}C_6} &\parallel \langle 100 \rangle_\gamma. \end{aligned}$$

Table 1. Chemical analysis of steels.

Steel	Composition, wt. %							
	C	Ni	Cr	Mn	Mo	Si	Nb	B
A	0.014	11.5	17.3	1.15	2.2	0.56	—	—
B	0.057	15.8	16.2	1.12	1.84	0.41	0.77	0.0003

Balance
„

1

2

3

Figures 1-3. Steel A, aged for 28.8 Ksec (8 hr) at 1073 K. Lamellar $M_{23}C_6$ formed close to incoherent twin boundaries and on coherent twin boundaries. 2. Steel A, aged for 1.80 Ksec (50 hr) at 1073 K. $M_{23}C_6$ plates increased in number on both sides of the incoherent twin boundary. The specimen foil is nearly parallel to the (110) of the matrix and the twin axis is $[111]$. 3. Steel B, aged for 14.4 Msec (4000 hr) at 1073 K. $M_{23}C_6$ plates formed on coherent twin boundaries and adjacent to incoherent twin boundaries, but considerably less in number.

specific features of the formation of these $M_{23}C_6$ lamellae were observed to vary from one twin to another.

The lamellar $M_{23}C_6$ precipitates, as reported by the previous investigators (Beckitt and Clark 1967; Singhal and Martin 1967) were assumed to result from emanation into the grains of the precipitates nucleated earlier, on incoherent twin boundaries. On the basis of this assumption in the mechanisms suggested for their formation the glide of the twinning dislocations possibly repelled from the incoherent twin boundaries by the stress field of growing particles at the boundaries has been proposed to play the most important role. The plate morphology has been attributed to result by a repeated nucleation (Beckitt and Clark 1967) on these gliding Shockley particles, or by the formation of stacking faults, nucleation on the outer particals and subsequent growth across the stacking faults (Singhal and Martin 1967). A close examination (by tilting the thin foils about different axes) of these lamellae, however revealed that most of them stood separate from the precipitates formed on incoherent twin interfaces, having no contact whatsoever. On the other hand, if any one of these mechanisms were operative, the lamellar $M_{23}C_6$ might not form in B since dislocations are more preferable sites for precipitation of NbC. Further, none of these mechanisms is able to explain the lamellar precipitation on coherent twin boundaries.

To examine further the validity of these models another experiment was made. A few bulk specimens of A were held at 173 K for sufficiently long periods, after being transferred directly from the solution-treatment temperature. As shown in figure 5, $M_{23}C_6$ precipitated in these specimens only on incoherent twin boundaries. No lamellar precipitation occurred either on coherent twin boundaries or in the regions adjacent to the incoherent twin boundaries. These observations indicated that quenching from the solution-treatment temperature is essential to induce lamellar precipitation during aging, and the existing models have no provisions to accommodate this fact.

Due to differential contraction between cooler and hotter parts of a metallic body, quenching from a high temperature induces pronounced stress. The state of stress inside a specimen becomes complicated in the vicinity of a barrier such as grain boundaries, twin boundaries and undissolved particles. The effect of the quenching stress near twin boundaries would, however, be different from that near a grain boundary owing to the inherent difference in characteristics of these two types of boundaries. In this respect coherent twin boundaries would once again behave differently from incoherent twin boundaries. Quenching stress produces residual stresses in the specimens, the maximum value of which has been calculated (Lang 1965) to be

$$\sigma_{\max} = \frac{E\alpha\Delta T}{1-\nu} \cdot \frac{S^2}{3at_0},$$

where E = Young's modulus; α = coefficient of thermal expansion; ΔT = temperature range ($T_q - T_{\text{room}}$); ν = Poisson's ratio ($= 0.3$); $2S$ = thickness of the plate; a = thermal diffusivity in cm^2/sec ; t_0 = total transient time.

The change of orientation of a crystal in a specific manner across twin boundaries is expected to induce a specific stress field around the boundaries since the elastic constants of a material are different along different directions. In these regions, $M_{23}C_6$ which always nucleates on $\{111\}$ austenite planes, because of least misfit (Beckitt and Clark 1967) across these interfaces has to choose one of the possible habits in order to

Figure 5. Dark-field optical micrographs of steel A. **a.** Water quenched and aged at 1073 K for 1.8 Msec (500 hr). $M_{23}C_6$ precipitated on incoherent and coherent twin boundaries and also in the regions adjacent to incoherent twin boundary. **b.** Quenched to 1073 K and held for 1.8 Msec (500 hr). Coherent twin boundaries and regions close to the incoherent twin boundaries are free from lamellar $M_{23}C_6$.

minimise the coherency strain. This is in conformity with the observations made. It is therefore, proposed that the presence of a residual specific stress field around twin boundaries in the quenched specimens is responsible for the formation of lamellar $M_{23}C_6$ precipitates on coherent twin boundaries and in the regions close to incoherent twin boundaries. Possibly vacancies play an important role in the process of their nucleation.

4. Conclusions

The lamellar $M_{23}C_6$ precipitates on coherent twin boundaries and near incoherent twin boundaries form during aging in the quenched samples only. The morphology and the specific orientation of these precipitates are influenced by the residual stress developed around twin boundaries due to quenching. The nucleation of these precipitates is aided by vacancies.

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