Displacive Phase Transformation and Surface Effects associated with Confocal Laser Scanning Microscopy

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Abstract

The influence of the free surface on martensitic transformation was examined by comparing the highest temperature at which martensite forms (M_S), as measured using dilatometry, with surface–observations using confocal laser scanning microscopy. It is found that the proximity of the surface during confocal microscopy permits martensitic transformation to occur at a higher temperature, with a reduced free energy change. This is because the strain energy due to the shape deformation accompanying the growth of martensite is reduced at a free surface. The second observation is that plates of martensite tend to coalesce as they approach the free surface where there is reduced constraint. The general observations are backed by calculating the strain-energy due to a sub–surface edge dislocation as a function of the orientation of its Burgers vector relative to the free surface.

1. Introduction

Confocal laser scanning microcopy (CLSM) is a powerful method for the study of phase transformations at high temperatures since the method avoids the deterioration of the image due to the thermal radiation [1]. However, observations using this technique may be influenced by the associated lack of constraint at the free surface, especially in the case of transformations which are dominated by strain energy. The martensitic transformation is such a case the strain energy resulting from the related shape deformation [2] is a seminal feature which determines not only the shape of the plate [3] but also the magnitude of the free energy change required in order to drive

the transformation [4]. Much of the reported literature in which confocal microscopy has been applied to steels has focused on the nucleation site or the evolution of the growing plate *etc.* in the context of martensite [1, 5, 6]. However, there is a dirth of systematic work to understand the surface effect itself and this was the aim of the present work.

Surface martensite has previously been studied in nickel–rich alloys [7–9]. An important interpretation about surface effect was in the work of Klostermann and Burgers [8] where it was concluded that in the Fe–Ni alloy studied, surface martensite forms at a temperature which is 5–30°C greater than that forming in the bulk of the material, because of the reduced constraint at the surface. The authors talked about hydrostatic pressure, but it is the relief of the much greater shear strains that should in fact dominate over volume change effects. We examine therefore, how a orientation of a plate whose growth is accompanied by a large shear, is influenced by the presence of a free surface.

2. Experimental Details

The work presented here is based on a more extensive study on the coalescence of plates of martensite or bainite, a process that leads to a deterioration in mechanical properties [10]. It was in the course of those studies that the alloy listed inTable 1 was studied using confocal laser scanning microscopy.

Table 1: Chemical composition of the alloy (wt%)

C	Si	Mn	Cr	Ni	Mo
0.03	0.23	2.05	0.43	7.1	0.63

Specimens for dilatometry were machined into 3 mm diameter cylinders of 10 mm. The specimens for CLSM were machined into $\approx$2 mm cubes. The details about the equipment are described in [10] and [11], respectively. In confocal laser scanning microscopy, the sensed temperature which controls the heat treatment differs from that of the specimen surface due to the heat transfer through the body of the alumina crucible on which the specimen is located, and the specimen itself. All the temperatures recorded during confocal microscopy as reported here are therefore corrected by calibration. The calibration was carried out by fixing a thermocouple to the top surface of

the specimen and comparing against the sensed–temperature of the confocal microscope furnace. The austenitization was carried out at 1250 °C for 3 minutes for dilatometry and CLSM. In the former case, the martensite-start (M_S) temperature was measured using the procedure of [12]. In CLSM, it was measured by determining the temperature at which surface relief due to the displacive nature of the transformation was observed. The severe problem of surface oxidation was avoided by maintaining an argon/helium atmosphere in the furnace, also containing 2 % hydrogen gas; titanium particles were also arranged around the specimen to induce cathodic protection.

The microstructures developed in the dilatometric and CLSM samples were also investigated using field emission gun scanning electron microscopy (FEGSEM).

3. Results
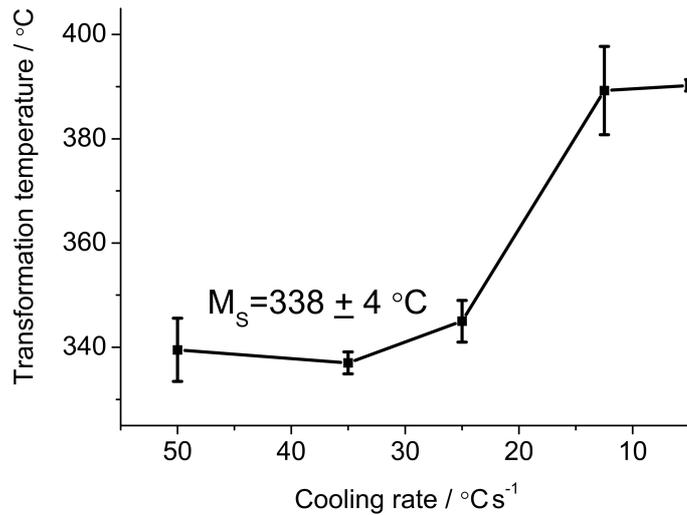
Figure 1: The variation of transformation temperature with cooling rate

The classical way of determining the martensite–start temperature is to cool the austenite at every increasing rates until the observed start–temperature becomes insensitive to the cooling rate. Fig. 1 shows the variation of transformation temperature with cooling rate in the dilatometric

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experiments. The transformation temperatures observed to be independent of cooling rate within the limits of experimental error were averaged to determine the M_S temperature, estimated as $338 \pm 4\,°C$.

One example of the surface relief observed during the transformation is illustrated in Fig. 2. The arrow indicates the temperature at which surface relief is first observed, corresponding to the initiation of transformation. This gives a start–temperature as $452 \pm 10\,°C$, where the error is estimated from experiments repeated five times.

In Fig. 3, the microstructure produced by confocal microscopy was compared that of the martensite produced by dilatometry. Notice that the microstructure for CLSM was taken from the cross section of the top and bottom surfaces, where the force on bottom surface due to specimen weight was neglected. It is odd that a typical lath structure of martensite was not observed near the top surface. On the other hand, the structure near the bottom surface corresponds to that of martensite from dilatometry. In addition, it was confirmed that (Fig. 4) that the top and bottom regions of the specimen were essentially cooled identically in terms of cooling rate, which is critical to identifying martensitic transformation. Therefore, the structure near the bottom surface was characterised as martensite, which formed not simultaneously but at the same temperature as that of top surface.

4. Discussion

As pointed out previously, Klosterman and Burgers [8] reported that in an Fe–Ni alloy, surface martensite forms some 5 to $30\,°C$ above the M_S temperature which applies to bulk martensite. In our case, this difference is significantly larger at $114 \pm 14\,°C$. Therefore, a previously developed program [13, 14] was used to analyse the thermodynamic effect of the increase of M_S temperature. Fig. 5 shows the change of chemical free energy of the alloy listed in Table 1 when it transforms from austenite to ferrite during cooling, without any change in composition. It is evident that the onset of martensitic transformation observed at the surface using confocal microscopy requires less driving force, where the reduction is compatible with the strain energy of elastically accommodated martensite plate, of $\approx 600\,J\,mol^{-1}$ [2, 4].

The relief of the strain energy due to martensitic transformation was investigated further by simulating the process as the interaction of a single

(a)

452 ℃   0s

10μm

(b)

435 ℃   3s

(c)

423 ℃   5s
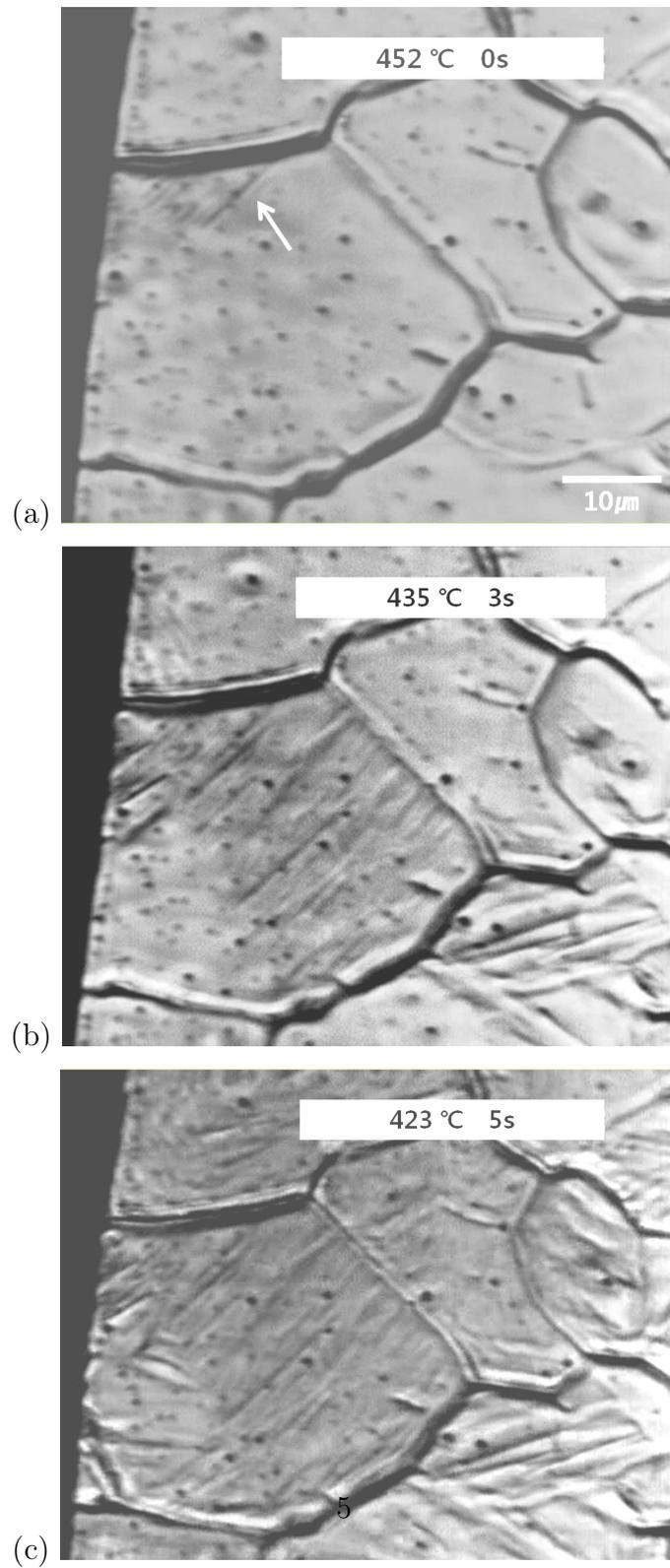
Figure 2: The surface relief that indicates phase transformation of austenite, as observed during confocal laser scanning microscopy.
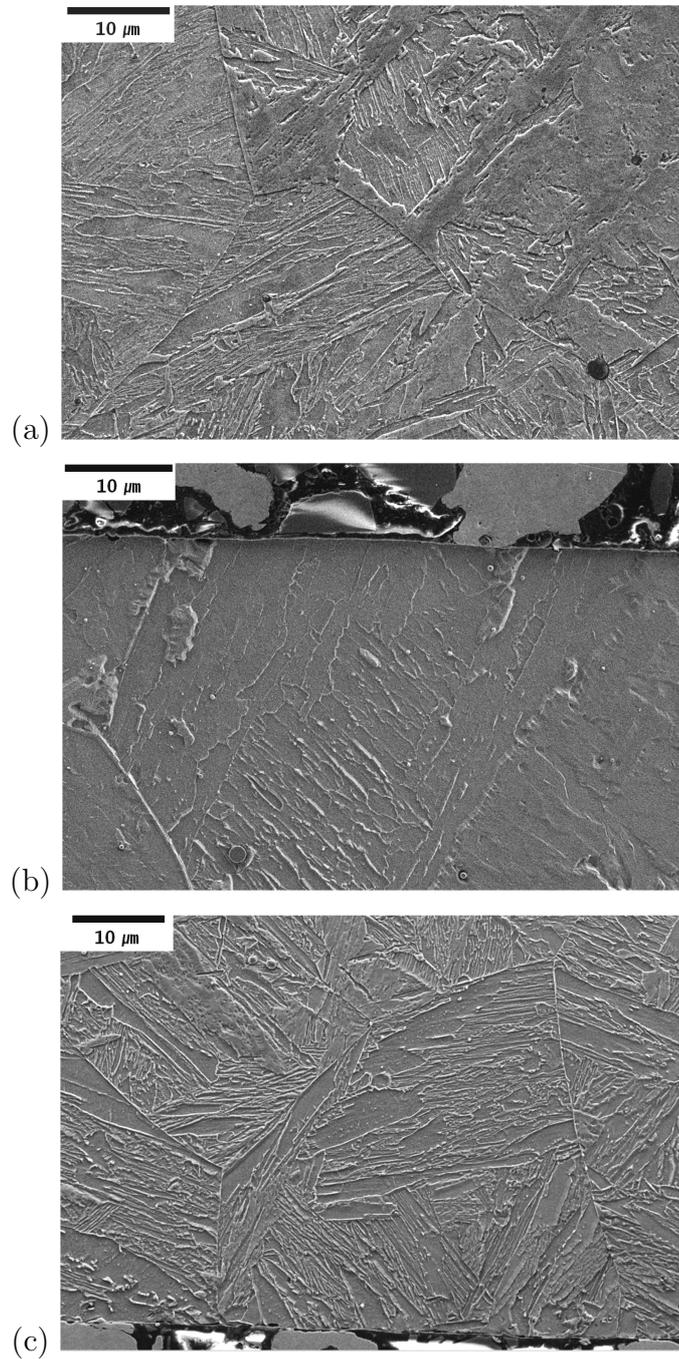
Figure 3: Microstructure generated in (a) dilatometry. (b) Confocal microscopy from the cross section of the top surface of the cube sample, and (c) the bottom surface.
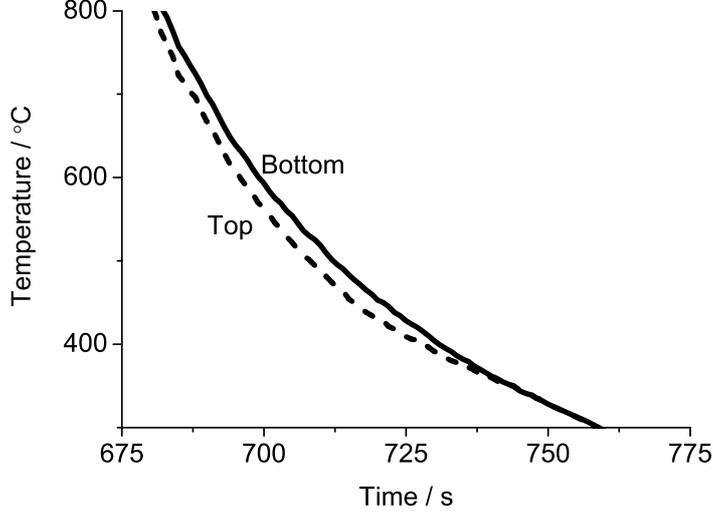
Figure 4: Cooling curves recorded near the top (dashed line) and bottom (solid line) parts of the specimen.

edge dislocation as a function of distance, and the angle between the Burgers vector and normal to the free surface. After all, the shape deformation involved in martensite transformation is dominated by a shear on the habit plane, as is the deformation due to an edge dislocation. We are using the shear dominated strain field of edge dislocations to represent the shear dominated shape deformation of martensite in order to simulate the influence of the free surface.

The Burgers vector of the dislocation was assumed to be parallel or normal to the free surface to examine the most favourable orientation that a martensite plate can adopt relative to the surface. First, the stress field caused by the dislocation was obtained from the work of [15]. This was substituted to following equation for the strain energy per unit length of the dislocation [16]:

$$\frac{W}{L} = \int_{r_0}^{R} r \, dr \int_{0}^{2\pi} \left[\frac{1}{2\mu}\sigma_{xy}^2 + \frac{1}{2E}(\sigma_{xx}^2 + \sigma_{yy}^2 + 2\nu\sigma_{xx}\sigma_{yy} - \sigma_{zz}^2) \right] d\theta \qquad (1)$$

where σ_{ij} is a stress component defined as a conventional way in [16]; E, μ and ν are Young's modulus, shear modulus and Poisson's ratio (taken to be 0.25),

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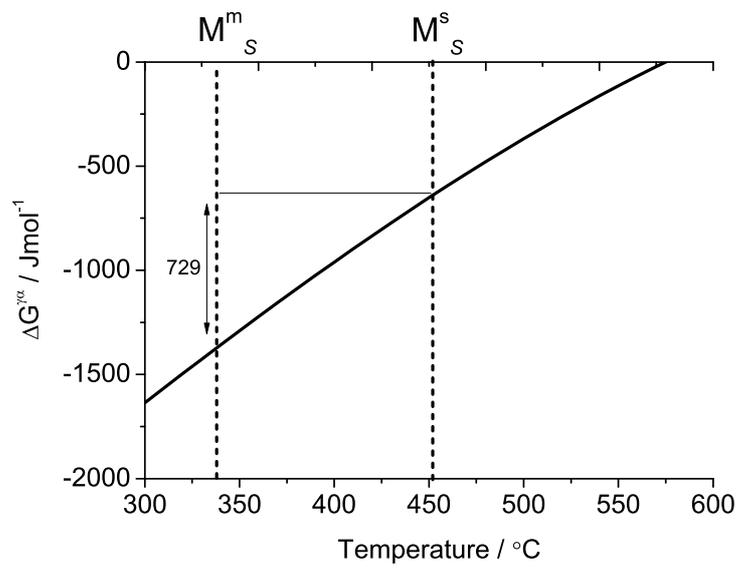
Figure 5: The chemical free energy change when austenite transforms into ferrite without any change in chemical composition, as a function of temperature for the alloy listed in Table 1. M_S^m and M_S^s represent the martensite-start temperatures in the bulk (dilatometric) and at the surface (confocal microscopy), respectively.

respectively. The terms r_0 and R are the length scales over which the energy density was to be integrated; r_0 was taken to be 4.5 times of the magnitude of the Burgers vector for a $\frac{1}{2}[110]$ dislocation on a $\{111\}$ plane in austenite [16] and $R = 5\,\mathrm{nm}$, which is a small enough value to show the characteristic behaviour. The dislocation core energy was neglected in the calculations due to its negligible contribution [16]. As expected, the calculations indicate that the strain energy of the dislocation decreases significantly as it approaches the surface, Fig. 6. However, the degree of the reduction is stronger for the dislocation related to the plate vertical to the free surface, which also might be expected if the shear is able to operate normal to the surface. This is in apparent contradiction with the notion that a plate lying nearly parallel to a free surface will experience a maximum strain-energy reduction [17], because that calculation implies that the plate forms a continuous surface layer, whereas in the present work the dislocation is located below the surface, simulating a plate that nucleates below the surface.

An interesting direct observation is that the martensite plates in Fig. 7 deviate in their growth direction to be vertical to the surface as they approach the surface. This provides experimental evidence that martensite plates originating from below the free surface prefer to be normal to that surface.

It is also noticeable in Fig. 7 that the martensite plates near the surface tend to coalesce. In fact [22] pointed that large strain energy must be endured for the coalescence. Since the surface reduces the strain energy associated with the martensite transformation as in Fig. 5 and 6, this represents a good environment to get coalescence.

5. Conclusions

Consistent with previous work, martensitic transformation is observed to form at a smaller undercooling when it occurs in the proximity of a free surface, when compared with that which forms constrained within the bulk of the material. The transformation is nevertheless thermodynamically consistent in that it occurs below the T_0 temperature where austenite and ferrite of identical composition have the same free energy. Furthermore, the calculated difference in free energy of transformation within the bulk and in the proximity of the free surface is consistent with the elastically accommodated strain energy due to the shape deformation of martensite.

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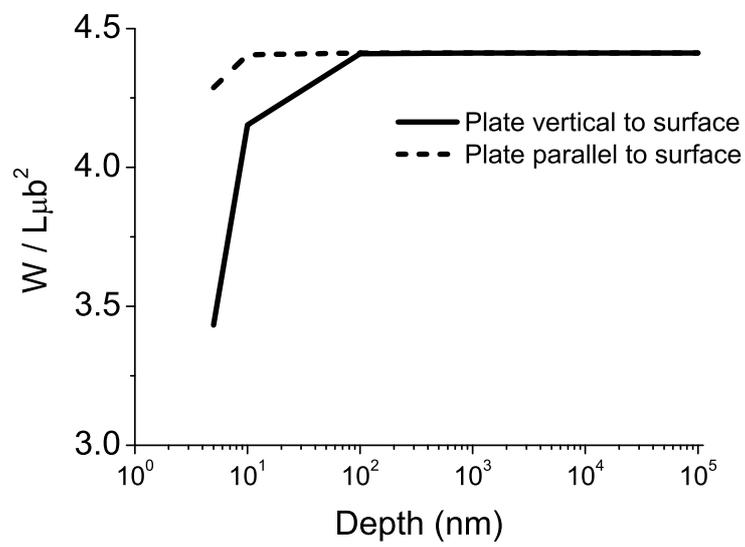
Figure 6: The variation of the strain energy due to an edge dislocation as a function of the distance from the surface. Solid and dotted lines correspond to the martensite plate which is vertical or parallel to free surface, respectively. The effect of the orientation of the Burgers vector becomes negligible when the dislocation lies deep within the material.
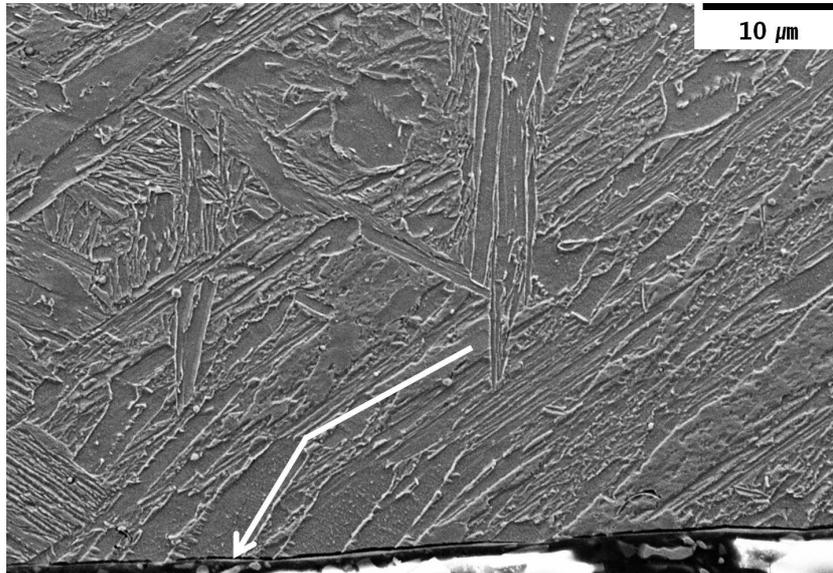
Figure 7: The martensite structure observed in the cross section of bottom surface. The arrow indicates the change of martensite-plates direction as they formed near the surface.

It has been demonstrated both by experimental observation and by simulation that the favoured orientation of a plate originating slightly below the surface will be such that its displacement vector is parallel to the surface normal. Other consequences of the presence of a free surface during martensitic transformation is that independent plates can coalesce as they approach the surface since the increase in the thickness to length ratio can be tolerated there due to the reduction in strain energy.

Acknowledgments

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