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An in situ transmission electron microscope study of the thermal stability of near-surface microstructures induced by deep rolling and laser-shock peening

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Abstract

We investigate the thermal stability of near-surface microstructures induced by deep rolling and laser-shock peening in AISI 304 stainless steel (AISI 304) and Ti–6Al–4V using in situ transmission electron microscopy. The improvements in fatigue resistance at elevated temperature are related to the high-temperature stability of the work-hardened near-surface microstructure.

Keywords: Ti–6Al–4V; AISI 304; Fatigue; Surface treatment; Deep rolling; Laser-shock peening

1. Introduction

Mechanical surface treatments, such as deep rolling, shot peening and laser-shock peening have seen widespread application in the automotive and aircraft industries owing to their beneficial effect on the fatigue resistance of metallic materials. The role of such treatments is primarily to induce near-surface compressive residual stresses, which serve to retard fatigue-crack propagation, and near-surface work-hardened nanoscale microstructures, which serve to retard fatigue-crack initiation by limiting plastic deformation. However, these mechanisms are only effective in service if they remain stable in the presence of cyclic and/or thermal loading. Although the stability of the residual stresses in mechanically surface-treated materials has been well studied, e.g., [1,2], little information exists on the corresponding stability of near-surface nanostructures induced by such treatments. Indeed, these near-surface regions have not been characterized by transmission electron microscopy (TEM) during, or even after, thermal exposure, despite the fact that such information is crucial for the higher-temperature use of mechanically surface-treated alloys. Since fatigue cracks are likely to initiate within these surface layers, it is important to characterize the

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nature and thermal stability of these regions. Accordingly, in the present work, we use cross-sectional TEM [3] and in situ heating of the TEM-foils to characterize the surface regions of mechanically surface-treated metals to determine the maximum temperatures at which these near-surface microstructures remain stable. The study focuses on two materials, AISI 304 stainless steel and Ti–6Al–4V, and two surface treatments, deep rolling (DR) and laser-shock peening (LSP), both of which are considered to be superior to shot peening for fatigue life enhancement [4].

2. Experimental procedures

The materials investigated were deep-rolled and laser-shock peened AISI 304 austenitic stainless steel and deep-rolled Ti–6Al–4V. The stainless steel was received from Schulte and Schmidt GmbH (Kassel, Germany) as solution treated and quenched, rolled cylindrical bars (14 mm diameter). To avoid grain-boundary precipitation, samples were not annealed prior to surface treatment and TEM-characterization. The microstructure was fully austenitic, with an average grain size of 150 μm, with a small volume fraction of chromium carbides and manganese sulfides. Uniaxial tensile testing at 25 °C gave a yield strength of 440 MPa, a tensile strength of 720 MPa, with 65% elongation.

The Ti–6Al–4V alloy originated as 63.5 mm diameter forging stock from Teledyne Titanium (Pittsburgh, PA), which was preheated at 940 °C and forged into $400 \times 150 \times 20$ mm plates [5]. The plates were solution-treated (925 °C, 1 h), fan-air cooled and stress relieved (700 °C, 2 h). The resulting bimodal microstructure consisted of interconnected equiaxed primary-α grains (64% by volume) and lamellar $\alpha + \beta$ colonies (transformed-β). The average α-grain size was ~20 μm; the average α-lath spacing was 1–2 μm. Room temperature tensile tests gave a yield strength of 930 MPa, a tensile strength of 978 MPa, with 45% reduction in area.

Deep rolling was carried out with a so-called ball-point rolling device, using a hydrostatic spherical rolling element (6.6 mm diameter), which permits the deep rolling of non-rotationally symmetric samples at a constant rolling pressure [6]. For both materials, a rolling pressure of 150 bar was applied (corresponding to a rolling force of 0.5 kN). Laser-shock peening was carried out using two overlapping layers of shocks (200% coverage), which were applied simultaneously to both specimen sides using 2.6×2.6 mm laser beams in 18 ns pulses with a fluence of 7 GW/cm^2.

Fatigue testing on both virgin and surface-treated material was performed under load control in tension–compression with a load ratio (minimum to maximum loads) of $R = -1$ using "smooth-bar" cylindrical samples, cycled at 5 Hz, with stress amplitudes of approximately half the tensile strength, at temperatures from ambient to 550–600 °C, i.e., $T/T_m \sim 0.4$–0.5 (T_m is the melting temperature), respectively, in the Ti–6Al–4V and AISI 304 [7–10].

Transmission electron microscopy was performed using a 300 kV JEOL microscope on cross-sections cut perpendicular to the surface of the surface-treated specimens; details of TEM-preparation methods are given elsewhere [9]. The effect of temperature was investigated by heating specimens, for durations between 5 and 20 min, in situ in the TEM from 25 to 900 °C. This was performed using a 300-kV JEOL 3010 TEM, equipped with a Gatan 652 double-tilt heating holder and a Gatan 794 Slow-Scan CCD for imaging. The sample holder temperatures were calibrated in a separate vacuum system to ensure accuracy to ±20 °C. Bright field images taken were from relatively thick sample areas so as to alleviate concerns of sample surfaces altering the grain-growth behavior. It should be noted that at the high magnifications used, the images only show the mechanically affected region.

3. Results and discussion

Fatigue results on virgin and DR material in Fig. 1 clearly show that there is a beneficial effect of mechanical surface treatment in prolonging the fatigue life of both alloys at temperatures as high as 600 °C ($T/T_m \sim 0.5$) in AISI 304 and 550 °C ($T/T_m \sim 0.4$) in Ti–6Al–4V. This is despite the fact that the compressive residual stress states induced

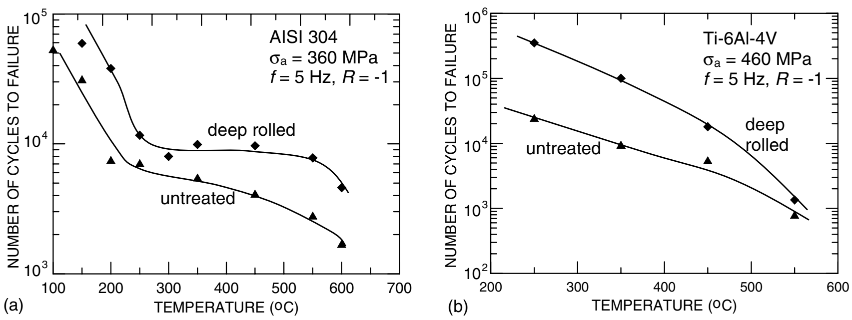

Fig. 1. Fatigue lifetimes at $R = -1$ for: (a) AISI 304 stainless steel [8] and (b) Ti–6Al–4V [10] in the untreated and deep-rolled state, at applied stress amplitudes, σ_a, of approximately half the tensile strength, i.e., 360 and 460 MPa, respectively, as a function of temperature of up to 550–600 °C. (*f* is the cyclic frequency).

in the surface layers have almost completely 'annealed-out' in both alloys at these temperatures [7,8]. This suggests that the near-surface microstructure plays a more prominent role in improving the fatigue life at elevated temperatures [10].

Both alloys displayed characteristic surface-treated microstructures in their near-surface layers following DR or LSP [10,11]. For AISI 304, deep rolling led to a complex subsurface structure of nanoscale austenitic and martensitic grains directly beneath the surface, extending 1–2 μm in depth (Fig. 2). This nanocrystalline layer, which is induced by severe plastic deformation and recrystallization, typically contained grains of 20–100 nm in diameter. In deeper regions, deformation bands consisting of inhomogeneous dislocation arrangements could be detected. Deeper still, i.e., >5 μm from surface, martensitic twin lamellae in an austenitic matrix could be observed within dense dislocation networks. In contrast to DR, laser-shock peening did not induce any near-surface nanocrystallization, but rather highly tangled and dense dislocation arrangements could be seen in the near-surface regions (Fig. 3). It is believed that the distinctly different microstructures are a consequence of the ~3–4 orders of magnitude higher strain rate involved in laser-shock peening [12].

The near-surface microstructures of deep-rolled Ti–6Al–4V also show a nanocrystalline grain structure (Fig. 4), similar to that in AISI 304. However in contrast to the steel, the grain size distribution is broader, with a locally inhomogeneous and high dislocation density, akin to that seen in the first stages of nanocrystallization in ball-milled metals [13].

To investigate the thermal stability of the near-surface microstructures, the TEM-foils were slowly heated in the microscope and observed at several temperatures after a holding time of ~5–10 min. For deep-rolled AISI 304, the near-surface nanocrystalline grain structure remains thermally stable up to 600–650 °C ($T/T_m \sim 0.5$), whereupon recrystallization commences and the average grain size is markedly increased (Fig. 2). For the corresponding laser-shock peened AISI 304, where a highly tangled and dense dislocation substructure forms rather than a nanocrystalline grain structure, the near-surface dislocation tangles were stable up to ~800 °C ($T/T_m \sim 0.6$). Above ~800 °C, the high dislocation density 'anneals out' and is replaced by the formation of low-angle grain boundaries (Fig. 3).

The near-surface nanostructure in deep-rolled Ti–6Al–4V appears to be perfectly stable up to 650 °C ($T/T_m \sim 0.2$–0.5) (Fig. 4) and for short durations up to 900 °C. Below 600 °C, increased holding times of 5–20 min did not yield any visible microstructural changes.

Thus, the improved fatigue resistance of both mechanically surface-treated alloys at 500–550 °C, where relaxation of compressive residual stresses has occurred, appears to be related to the thermal stability of the work-hardened near-surface layers. Both the nanocrystalline grain size and high dislocation densities act as obstacles to stable dislocation slip, thereby increasing resistance to fatigue-crack initiation.

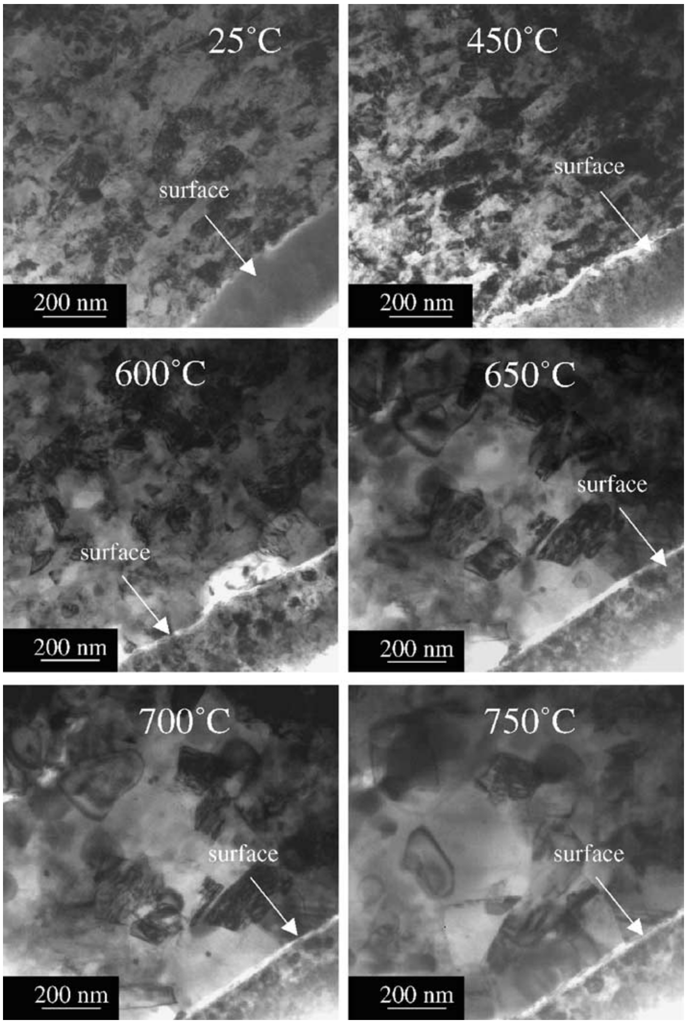

Fig. 2. Near-surface microstructure of deep-rolled AISI 304 (rolling pressure: 150 bar, spherical rolling element: ø6.6 mm) after thermal exposure at temperatures between ambient and 750 °C.

4. Conclusions

Based on an investigation into the short-term thermal exposure of near-surface microstructures induced by mechanical surface treatment, deep rolling and laser-shock peening, in AISI 304 austenitic stainless steel and a Ti–6Al–4V alloy, the following conclusions can be made:

(i) The beneficial effect of DR and LSP on the fatigue life at temperatures as high as 550–600 °C, where almost complete relaxation of residual stresses has occurred, appears to be related to the thermal stability of the work-hardened near-surface microstructures.

(ii) In AISI 304, a nanocrystalline grain structure forms in the near-surface layers after deep rolling which remains stable until it recrystallizes at ~600–650 °C. A highly tangled and dense dislocation substructure is formed near-surface after LSP, which is stable up to ~800 °C. Deep-rolled Ti–6Al–4V also forms a nanocrystalline near-surface structure with high dislocation density, which is stable up to 650 °C.

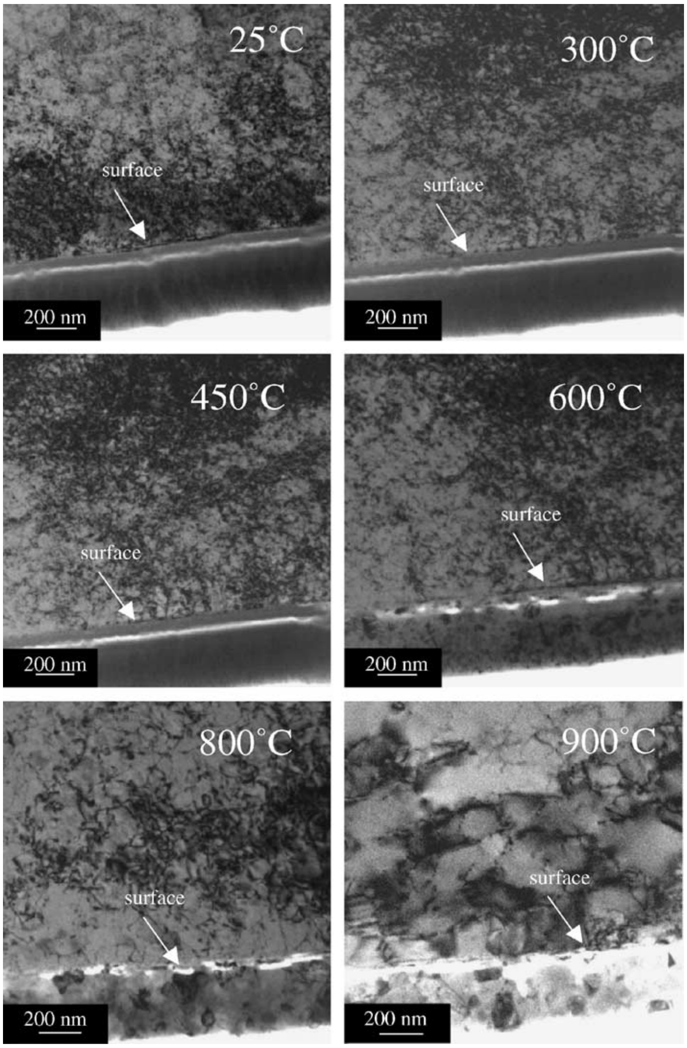

Fig. 3. Near-surface microstructure of laser-shock peened AISI 304 (intensity: 7 GW/cm^2, coverage: 200%) after thermal exposure at temperatures between ambient and 900 °C.

(iii) It is reasoned that the beneficial effect of these near-surface work-hardened regions is to inhibit dislocation glide through the nanocrystalline grain size and high dislocation density, thereby improving resistance to fatigue-crack initiation.

Acknowledgements

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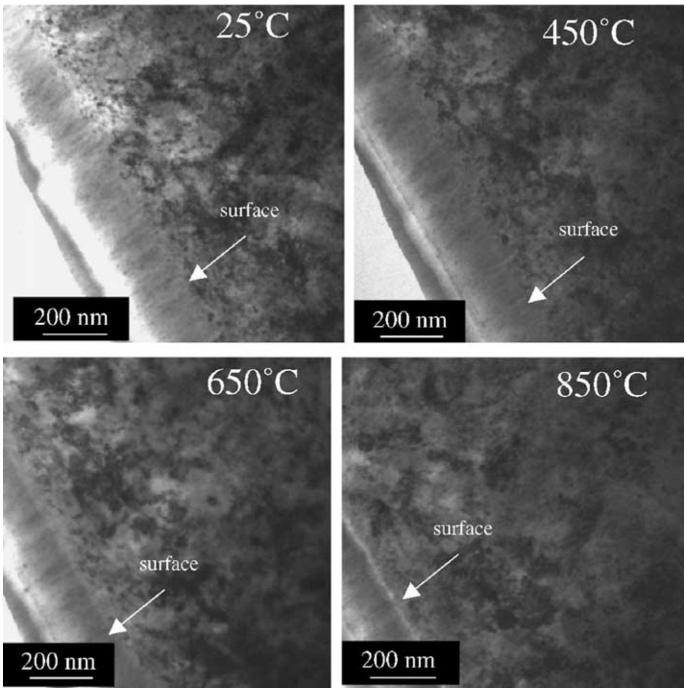

Fig. 4. Near-surface microstructure of deep-rolled Ti–6Al–4V (rolling pressure: 150 bar, spherical rolling element: ø6.6 mm) after thermal exposure at temperatures between ambient and 850 °C.

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