TECHNICAL NOTE

X-RAY FRACTOGRAPHY STUDIES ON AUSTENITIC STAINLESS STEELS

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Abstract—In this investigation, the fracture surfaces of SS 304 and SS 316 austenitic steels were analysed using the X-ray fractography technique. In both cases, a decrease in the austenite content was observed at the fracture surface as a result of deformation induced martensite, indicating a linear relation with $K_{\max}$ within the stable crack growth region. The presence of this martensite was found to be confined to a very thin layer close to the fracture surface. The magnitudes of the residual stress (σ_r) as well as the profile breadth (B) as a function of $K_{\max}$ in martensite were found to be higher than in austenite, with greater differences in SS 304 than in SS 316. In both the steels a gradual decrease of σ_r with depth below the fracture surface was noticed. While a gradual decrease of B was noticed in SS 304, a shallow minimum was noticed at a depth of about 100 μm in SS 316. Copyright © 1996 Elsevier Science Ltd.

1. INTRODUCTION

X-RAY FRACTOGRAPHY is a method in which a fatigue fracture surface is examined using the X-ray diffraction technique. The measured residual stress (σ_r) and the Full Width at Half Maximum (FWHM, B) are correlated to the fracture mechanics parameters such as maximum stress intensity factor ($K_{\max}$) or stress intensity factor range (ΔK). Since its development, this method has been employed by many investigators to analyse the fracture surfaces of fatigue specimens of different materials. Such analyses include X-ray stress measurements at and below the fracture surface. Attempts were also made to establish possible correlations between laboratory generated information and that obtained from fracture surface analysis of components failed in service. This method was found to be successful only in a few cases.

In the literature, so far, only one investigation [1] is cited concerning the X-ray fractography studies on fatigue fracture surfaces of SS 304 type austenitic stainless steels. In continuation to the earlier investigations on C45 grade ferritic steel specimens [2], the present work was taken up by the authors to examine the fracture surface in two different austenitic stainless steels viz. SS 304 and SS 316. The influence of deformation induced martensite on the residual stress (σ_r) and the Full Width at Half Maximum (FWHM, denoted by B) of the X-ray profile as a function of the maximum stress intensity factor ($K_{\max}$) as well as the depth below the fracture surface was investigated in these steels.

2. EXPERIMENTAL DETAILS

Austenitic stainless steels of grades SS 304 and SS 316 in the rolled and heat treated condition (as received) were selected. The chemical composition of the steels are shown in Table 1. The typical microstructures are shown in Fig. 1. The average austenite grain size was found to be about 60 μm (ASTM No. 5) in SS 304 steel and 30 μm (ASTM No. 7) in SS 316 steel.

Compact Tension (CT) specimens of 12.5 mm thick were prepared and tested for fatigue crack growth rate according to the ASTM E-647 standard. The fatigue tests were conducted on a 100 kN universal static/dynamic MTS servo-hydraulic test system using a stress ratio of 0.1 and a frequency of 20 Hz. The crack extension measurements were performed with the help of a low power travelling microscope and a stroboscope.

The fatigue fracture surfaces of the specimens tested, were examined in a high resolution Hitachi S-800 Scanning Electron Microscope (SEM) for fracture morphologies at different crack lengths. Subsequently, the X-ray residual stress measurements on these surfaces were performed at different crack lengths, using the standard multiple exposure $\sin^2\psi$ method [3]. A Rigaku Strainflex MSF-2M type portable X-ray stress analyser was employed for this purpose. All the residual stress measurements were performed using the (220) austenite reflection with a Cr K_α radiation. In addition, measurements on martensite were performed using the (211) reflection with the same radiation. The latter was found necessary due to the presence of the deformation induced martensite at the fracture surface locations as well as at depths of a few tens of

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Table 1. Chemical composition (weight %) of the steels investigated

Steel	C	Mn	Si	S	Ni	Cr	Mo
SS 304	0.06	1.65	0.33	0.005	8.82	17.72	0.40
SS 316	0.047	1.01	0.53	0.02	10.68	16.07	1.71

microns below the fracture surface. Sub-surface stress measurements were performed after successive layer removal of the material by electro-chemical polishing technique.

3. RESULTS AND DISCUSSION

Fatigue crack growth tests were performed on both grades of steels and the respective plots are shown in Fig. 2. The crack growth rates in both steels appear to be nearly identical. The "m" values (about 3.3) were found to be lower than those for C45 steels [4]. The plots are linear up to a ΔK value of about $30 \text{ MPa}\sqrt{\text{m}}$ for SS 304 and up to slightly more than $40 \text{ MPa}\sqrt{\text{m}}$ for SS 316. These appear to be the transition points from stable crack growth (stage II) to the onset of fast fracture (stage III). A significant necking was also noticed on sections of the test samples at locations where ΔK is more than about $40 \text{ MPa}\sqrt{\text{m}}$.

Representative fracture morphologies for SS 304 and SS 316 steels are shown in Figs 3 and 4, respectively. In general, the fractures in the stable crack growth region show quasi-brittle features [Figs 3(a) and 4(a)]. A transition from transgranular to intergranular is noticed in both the cases at a K_{max} value of about $30 \text{ MPa}\sqrt{\text{m}}$ [Figs 3(b) and 4(b)]. Typical fatigue striations are observed at locations corresponding to stage III region [Figs 3(c) and 4(c)]. At farther locations corresponding to the fast fracture regions ($K_{\text{max}} \geq 50 \text{ MPa}\sqrt{\text{m}}$) typical ductile fractures containing dimples are noticed [Fig. 3(d)].

Transformation of austenite to deformation induced martensite as a consequence of fatigue cycling effects at the crack tip was noticed in both cases. The martensite content ($\alpha\% = 100 - \gamma\%$) was estimated by comparing the integrated intensities of α (211) and γ (220) profiles. The amount of transformed martensite was found to increase with K_{max} and such a variation is shown in Fig. 5, for both steels. This is expected as the extent of deformation increases with K_{max} . This variation is approximately linear up to a K_{max} value of about $30 \text{ MPa}\sqrt{\text{m}}$ in SS 304 and up to $40 \text{ MPa}\sqrt{\text{m}}$ in SS 316. For SS 304 an increase from 40% to 60% is noticed as K_{max} increases from 20 to $30 \text{ MPa}\sqrt{\text{m}}$. At still higher K_{max} ($\geq 70 \text{ MPa}\sqrt{\text{m}}$), a much larger amount (about 80%) of martensite is observed. In SS 316 steel, an increase in martensite from ca 25% to 50% is noticed for a corresponding increase in K_{max} from ca 20 to $40 \text{ MPa}\sqrt{\text{m}}$. At the fast fracture locations for which $K_{\text{max}} \geq 40 \text{ MPa}\sqrt{\text{m}}$ the amount is ca 70%. The relatively lower transformation of austenite to martensite of SS 316 steel at any K_{max} may be attributed to the differences in the chemistry of the two alloys. The austenite of SS 316 steel is known to be more stable than that of SS 304 steel due to a higher ratio of austenite to ferrite stabilizing elements in SS 316.

Stress measurements were performed on both austenite and martensite phases at every location on the fracture surface. Variations in the residual stress (σ_r) in both phases as a function of K_{max} are shown in Fig. 6 for SS 304 and Fig. 7 for SS 316 steels. The stresses at any K_{max} are larger in martensite phase than those in the austenite phase. The largest difference (ca 400 MPa) is greater with SS 304 (Fig. 6) than with SS 316 (Fig. 7). While the stress differences in SS304 reduce with K_{max} (Fig. 6), such differences in SS 316 continue to remain nearly the same even in the fast fracture (higher K_{max}) regions.

The stresses in the deformed martensite of both steels and the austenite of SS 316 show an increasing trend in lower K_{max} regions up to ca $30 \text{ MPa}\sqrt{\text{m}}$ followed by a decreasing trend in higher K_{max} regions. On the other hand, the stresses

Fig. 2. Log (da/dN) vs Log ΔK plots for (a) SS 304 and (b) SS 316 steels.

Fig. 1. Typical microstructures of (a) SS 304 and (b) SS 316 steels (magn: 200 ×).

Fig. 3. Typical SEM fractographs for SS 304 steel.

Fig. 4. Typical SEM fractographs for SS 316 steel.

Fig. 5. Deformation induced martensite variation with K_{max} .

in the austenite of SS 304 show a continuously decreasing trend. The observed increasing trend at lower K_{max} regions could be attributed to the cumulative strain cycling effects, which increase with crack length or K_{max} . At higher K_{max} regions the monotonic loading effects become more prominent than cumulative cycling effects since the material ahead of the crack tip is subjected to larger plastic strain cycling, but for a smaller number of fatigue cycles. Normally the monotonic deformation will lead to the development of compressive residual stress the magnitude of which increases with the amount of deformation. As a result, the residual stress variation at higher K_{max} regions exhibits a decreasing trend to a minimum value. Such trends were also noticed in C45 [2, 4] and duplex stainless steel [4] fracture surface analysis. In addition to the monotonic deformation effects, the stresses which develop in the martensite when it transforms from austenite are likely to influence the residual stress distribution at the fracture surface. This transformation is accompanied by a volume increase which is constrained by the surrounding untransformed austenite, thus keeping the transformed martensite phase in compression. This compressive stress will counter the tensile stress developed in that phase due to fatigue deformation. With increasing K_{max} (increased amount of deformation) the amount of transformed martensite increases resulting in larger compressive transformation stresses. When the effects due to these transformation stresses and the monotonic deformation at higher K_{max} levels become greater than the fatigue deformation effects, even a net compressive residual stress could be expected.

Fig. 6. Residual stress distribution as a function of K_{max} for SS 304 steel.

Fig. 7. Residual stress distribution as a function of K_{max} for SS 316 steel.

Changes in FWHM (B) of both phases with K_{max} are shown in Fig. 8 for SS 304 and in Fig. 9 for SS 316 steels. The profile breadths (B) at any K_{max} from the martensite phase are much larger ($\cong 2$ deg.) than those from the austenite. The B variations exhibit a gradual increase with K_{max} . With an increase in K_{max} , there is more broadening of the martensite diffraction profile of SS 304 than that of SS 316. This could be related to the higher percentage of transformed martensite for SS 304 than for SS 316 (Fig. 5). The situation appears to be reversed for the austenite diffraction profile, though less prominent. For some reason, this increasing trend is not continued in the austenite phase of SS 316 steel beyond a K_{max} value of ca 60 MPa/√m.

In sub-surface analysis, the amount of transformed martensite was also measured at every depth of observation in addition to the residual stresses. The variation of the amount of martensite as a function of depth below the fracture surface is shown in Figs 10 and 11 for both steels. In both the cases, the martensite content decreases rapidly within a depth of ca 7 μm and reaches a zero level at depths of ca 12 μm for SS 316 and between 7 and 20 μm for SS 304. From this observation one can qualitatively say that in SS 304 the martensite is present to a larger depth than in SS 316, which is once again related to the fact that the austenite of SS 316 is more stable than that of SS 304.

The variations in residual stress (σ_r) in the austenite phase as a function of depth are shown in Figs 12 and 13, for both the steels. In both cases, a sharp decrease in σ_r occurs within 100 μm below the fracture surface. Beyond these depths a more gradual decrease is noticed up to ca 300 μm . Unlike the observations made in the fracture surface analysis of C45

Fig. 8. FWHM (B) distribution as a function of K_{max} for SS 304 steel.

Fig. 9. FWHM (B) distribution as a function of K_{max} for SS 316 steel.

and duplex steel specimens [4], no stress relaxation effects as revealed by the increasing trends of σ_r within a few tens of microns depth below the fracture surface were noticed for both the SS 304 and SS 316 steels. This indicates that the maximum relaxation of the stresses developed within the plastic zone (due to plastic deformation) occurs at the fracture surface itself. At larger depths, compressive stresses of the order -50 to -100 MPa were noticed. The observed scatter in residual stress values is due to grain size effects.

Corresponding breadth (B) variations with depth are shown in Figs 14 and 15. A sharp decrease is noticed within $100\ \mu\text{m}$, beyond which the variations are gradual. In SS 316 the trends indicate a shallow minimum *ca* $100\ \mu\text{m}$ below the surface, the reasons for which are not clear. In both the cases, B approaches a value of about 1 deg. at larger depths.

It may be noted that changes in B are sensitive to microstructural changes in a material caused by plastic deformation or recovery. As a consequence of plastic deformation ahead of the crack tip, generally, one can expect the development of a large number of dislocations in the deformed material. Their density and distribution will depend on the generation and annihilation rates which in turn are related to the microstructure, crystal structure, stacking fault energy, and type and number of obstacles. In fatigue cycling, the additional plastic strain cycling effects ahead of the crack tip could result in some dynamic recovery. Since all these effects are highest in close proximity to the growing crack, the recovery effects will also be higher at and near the fracture surface. The amount of deformation decreases with distance from the crack

Fig. 10. Deformation induced martensite as a function of depth for SS 304.

Fig. 11. Deformation induced martensite as a function of depth for SS 316.

tip. When dynamic recovery is considered, its contribution should be highest at the fracture surface where maximum deformation and possibly the greatest temperature rise occur. The recovery effects decrease with depth. Deformation and recovery effects will influence the FWHM (B) values significantly. While plastic deformation increases B , the recovery should decrease B . The B variations with depth at different K_{max} show a gradual decrease, with the highest B at the fracture surface itself. This indicates possibly no recovery, with the maximum deformation effects being on the fracture surface. Since the stacking fault energy is low for austenite, the recovery will be difficult as the recovery temperature will be relatively high. The reasons for the shallow minimum observed at a depth of ca 100 μ m below the fracture surface of SS316 steel specimen were not clear. Further studies are recommended for detailed analysis.

4. CONCLUSIONS

The deformation induced martensite content as measured at the fracture surface was found to vary linearly with K_{max} within the stable crack growth region, in both the austenitic steels. The amount of transformed martensite observed was larger in SS 304 than in SS 316 steel. This could be attributed to the higher austenite stability in SS 316.

Fig. 12. Residual stress distribution below fracture surface of SS 304 steel.

Fig. 13. Residual stress distribution below fracture surface of SS 316 steel.

The increasing trends of σ_r at lower K_{max} regions were attributed to the cumulative strain cycling effects, which increase with crack length or K_{max} . The observed decreasing trends at higher K_{max} regions were attributed to the increased monotonic loading effects. In addition to these effects, the decreasing trends observed in the case of the deformation induced martensite at higher K_{max} regions have been attributed to the compressive stresses which develop in the martensite when it transforms from austenite.

The deformation induced martensite in these steels is limited to a very thin layer of material close to the fracture surface (20 μm maximum).

The fracture morphologies indicate a transition from transgranular to intergranular mode within the stable crack growth region (stage II).

In the fracture surface of both the steels, the magnitude of residual stress (σ_r) as well as the profile breadth (B) of martensite were found to be higher than those of austenite. The largest differences were found in SS 304.

A significant decrease in σ_r and B values of austenite was observed within 100 μm below the fracture surface. This indicates the maximum possible relaxation and recovery effects, if any to occur, take place at the fracture surface itself.

Fig. 14. FWHM (B) distribution below fracture surface of SS 304 steel.

Fig. 15. FWHM (B) distribution below fracture surface of SS 316 steel.

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