2.5 TENSILE PROPERTY CHARACTERIZATION OF 14YWT NANOSTRUCTURED FERRITIC ALLOY NFA1 -

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OBJECTIVES

The main objective of this study is to characterize the high temperature tensile property of a newly developed larger best practice heat of 14YWT nanostructured ferritic alloy, NFA1, indifferent orientations.

SUMMARY

FCRD NFA-1 is a best practice nanostructured ferritic alloy (NFA) produced by ball milling argon atomized Fe-14Cr-3W-0.4Ti-0.2Y (wt.%) and FeO powders, followed by consolidation, extrusion, annealing and cross-rolling. Tensile tests in six orientations at temperatures 23 to 800°C exhibited excellent strength in the extrusion and cross-rolled directions (LT and TL), accompanied by total elongation of ~10%. In contrast, loading in the short plate thickness direction (S) at ambient temperature exhibited much lower strength with a flat, facet fractured surface, and total ductility of less than ~0.5%. The low strength and low total strain in the plate thickness direction can be attributed to the presence of pre-existing microcracks running parallel to the plate surfaces that propagate by brittle cleavage.

BACKGROUND

Background on NFA and further details on NFA-1 are reported elsewhere and will not be reviewed here [1,2].

EXPERIMENTAL

The as fabricated NFA-1 was characterized using a scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) and electron backscatter diffraction (EBSD). Polished (down to 20 nm colloidal silica) surfaces were observed in a FEI X30 SEM (Netherland) to characterize the surface morphology before testing. EBSD was used to measure the crystallographic grain morphology with respect to extrusion and cross-rolling directions. Tensile tests were performed on SSJ-2 type dog-bone shaped and sub-sized flat specimens with a gage section length, width and thickness of 5.0x1.2x0.5 mm. The tests in air were performed for six orientations, namely LT, TL, LS, TS, SL and ST where L, T and S represent longitudinal (rolling), transverse (cross-rolling) and short thickness directions, respectively (see Fig.1). The tests were conducted at varying temperatures from ambient to 800°C, using a 810 MTS servo-hydraulic universal testing machine equipped with a clam shell furnace. All specimens were sanded down to 1500 grit to remove any surface contamination or residual stresses due to the EDM machining before testing. The specimens were heated to the target temperature in atmosphere environment, and held for 10 minutes before testing. Testing was carried out at a crosshead speed of 0.30 mm/min or a strain rate of $\approx 10^{-3}$/s. Except for the small size of the specimens, the tensile properties were determined in accordance with ASTM Standard E8M-13. Extensive SEM/EDS was carried out on the post-test fracture surfaces.

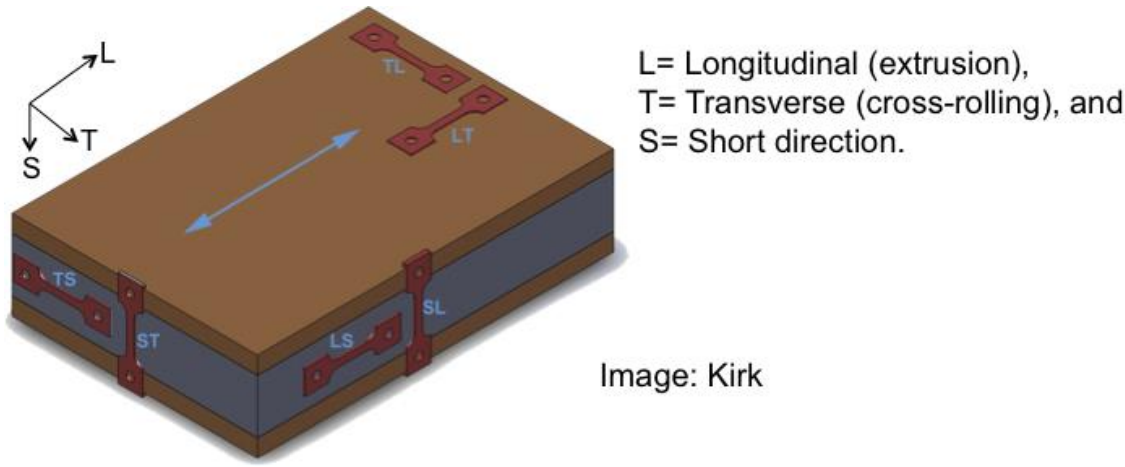


Figure 1. NFA1 specimen orientations labeled with respect to the extrusion, cross-rolling and plate thickness directions.

STATUS AND PROGRESS

Pre-Test Characterization

SEM micrographs on non-deformed polished and etched NFA-1 plate normal surfaces in Fig. 2a do not show any cracks or pores. In contrast, Figs. 2b and c show anisotropic banding in the thickness cross section plane oriented along cross-rolling (plane B) and extrusion (plane C) directions. EBSD used to characterize the grains imaged in all three planes. The inverse pole figure (IPF) maps in Fig. 3 highlight the existence of strong preferential crystallographic fiber texture, with most grains <110> oriented along the extrusion direction. Plane A showed the smallest average grain size of ≈ 235 ± 212 nm, with the lowest grain aspect ratio (GAR) of ≈ 1.7 ± 0.6. Fig. 3b shows the presence of surface cracks along the transverse direction. Most of the grains are extended along the transverse (cross-rolling) direction (Fig. 3b). Very large (2-5 micron) and elongated grains are often observed in the vicinity of the surface cracks. Plane C also reveals relatively larger 349 ± 247 nm grains with the highest GAR ≈ 3.3. Fig. 3c reveals that the elongated grains oriented along the extrusion directions are also often associated with surface cracks.

Tensile Properties

Room temperature tensile tests were conducted in six different orientations shown in Fig. 1. Examples of the results are shown in Fig. 4. The LT orientation showed the highest ambient temperature strength with a YS average of 1144 MPa 0.2% and UTS average of ≈ 1253 MPa, The corresponding ST oriented specimens showed the lowest average strength of ≈ 765 MPa and 832 MPa for the YS and UTS, respectively. The fracture surfaces in all six orientations are shown in Fig. 5. There is a similarity in properties and fracture patterns that fall three different categories: group 1 LT and TL orientations (group 1) with high strength and total strain; LS and TS (group 2) with the highest total strain; and SL and ST (group 3) with the lowest strength with negligible total strain and brittle fracture surfaces.

Delaminations occurred in the group 1 normal to the thickness direction in planes parallel to the plate surfaces.In group 2 delaminations form through the width direction along the gage length normal to the thickness direction, which is basically a 90° rotation from group 1. Stress-strain curves in Fig. 4 show multiple-stage post necking load drops associated with a sequence of delaminations. The load drops are due to the corresponding to reductions in the necking constraint in the width direction. Difference between group 1 and 2 are related to the brittle orientation of the brittle planes with respect to the direction of necking reduction. The LT/TL oriented specimens neck in the thickness direction whether as LS/TS oriented specimens neck in width direction. Specimens loaded in the short directions (group 3) failed almost in a brittle manner, mostly in its elastic regime. In this case the weak planes and laminated layers are perpendicular to the loading direction and easily break during tensile loading at much lower stress compared to other orientations. The Fractured surfaces are quite flat, in this case, with almost

no/minimal area reduction. Group 2 specimens exhibited the highest uniform and total elongations. The LS orientation exhibits an average uniform elongation (UE) of ≈ 8.1% and a total elongation (TE) is as high as 21.8%. Group 1 specimens exhibited lower total strain with averages of ≈ 5.0 % UE and ≈ 10% TE. The higher strength of group 1 specimens comes at the expense of total strain, compared to group 2 specimens. In contrast, group 3 specimens exhibited both lower strength and total strain (≤ 0.5% UE and TE) compared to the other groups. Figure 6 shows higher magnification SEM images of fractured samples that revealed the extensive presence of ductile shear zones in group 1 and ductile dimples in group 2 in contrast to the cleavage brittle fracture features in group 3 specimens.

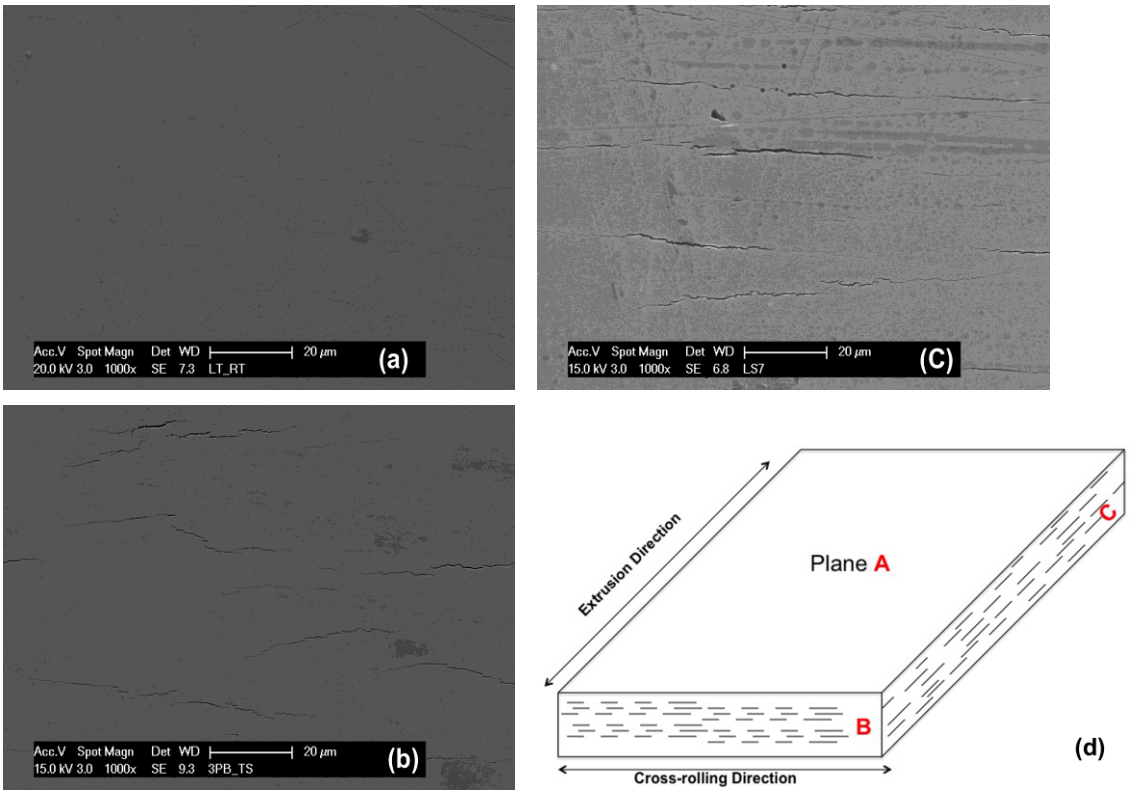


Figure 2. SEM images showing the non-deformed surface morphology of: (a) Plane A, (b) Plane B, and (c) Plane C of 2(d), respectively. 2(d): Schematics of delamination of NFA1.

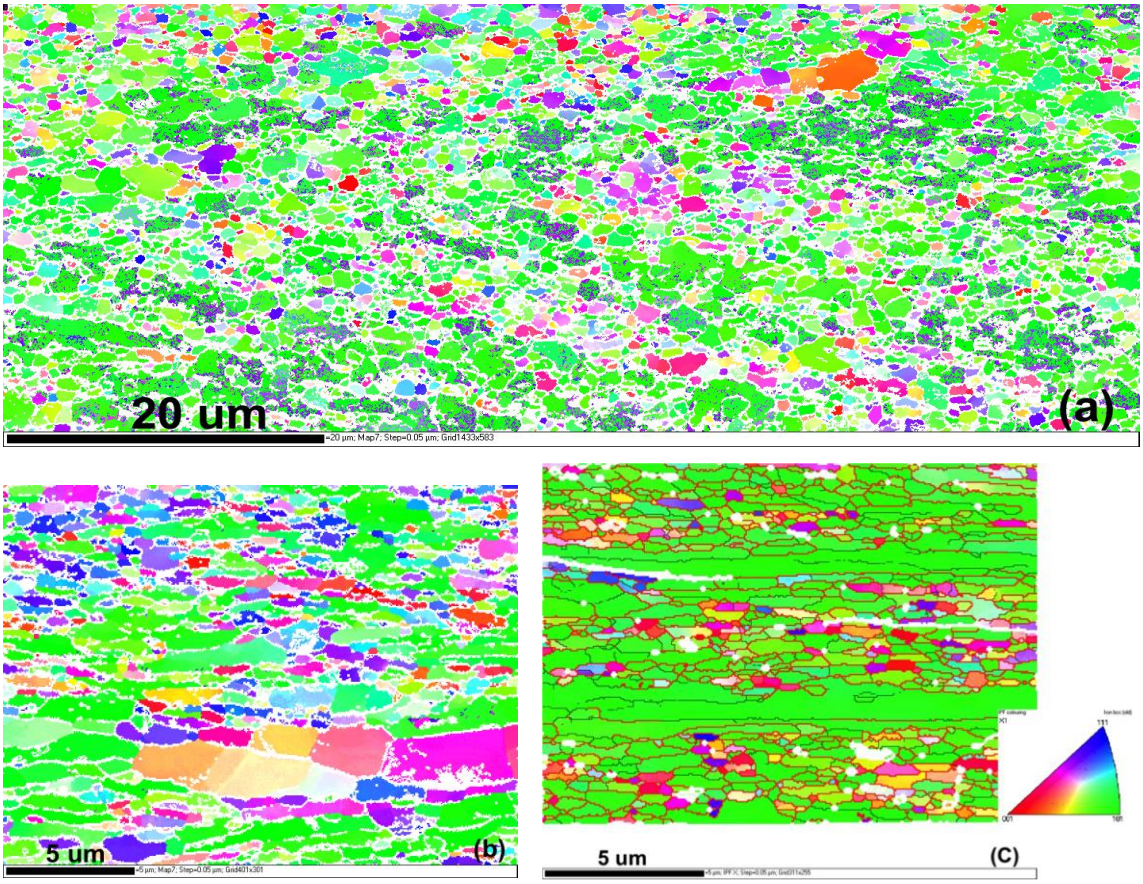


Figure 3. Inverse pole figure (IPF) maps obtained by EBSD on the: (a) Plane A, (b) Plane B, and (c) Plane C of 2(d), respectively.

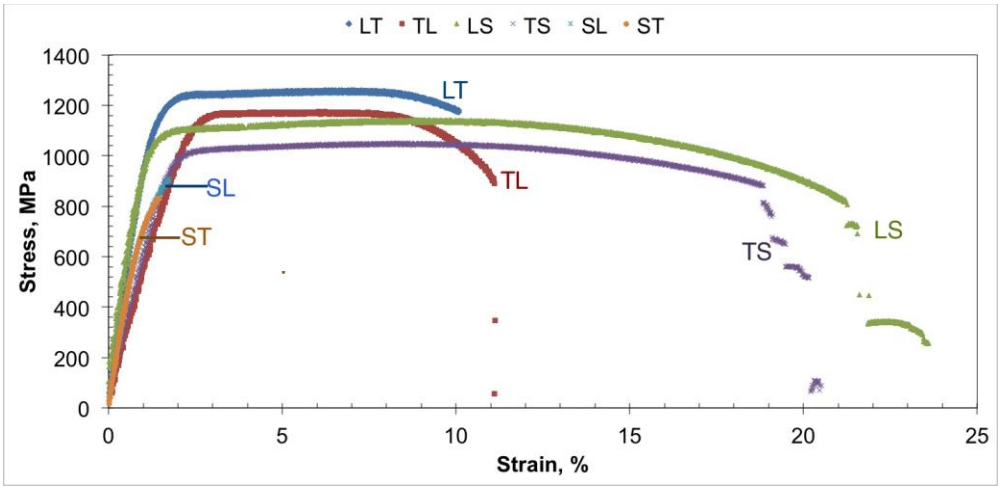


Figure 4. Room temperature engineering stress-strain curves of NFA1 specimens tested ain all six orientations.

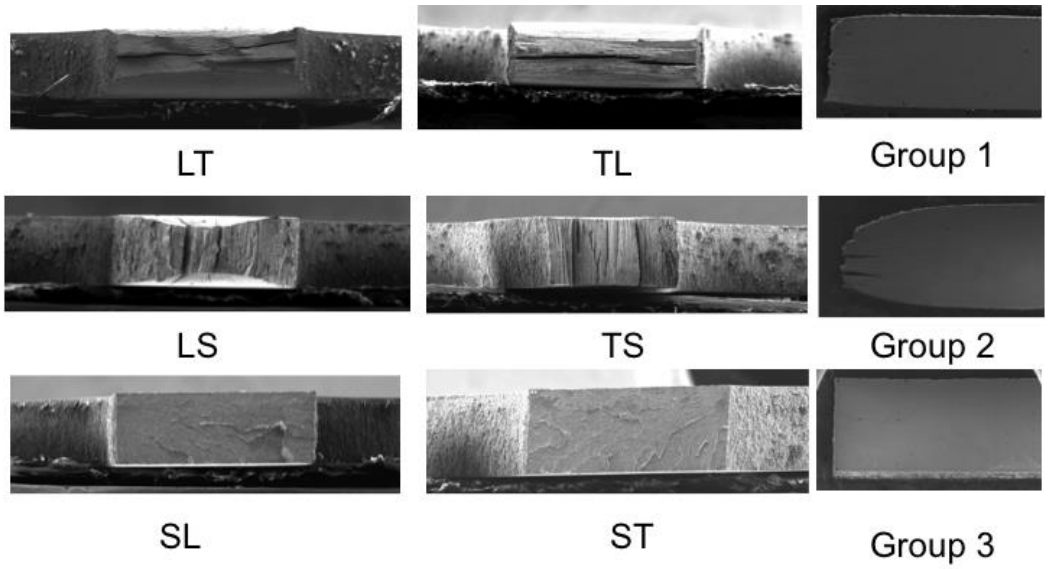


Figure 5. Low magnification SEM fractographs of room temperature tensile fracture surfaces in different orientations.

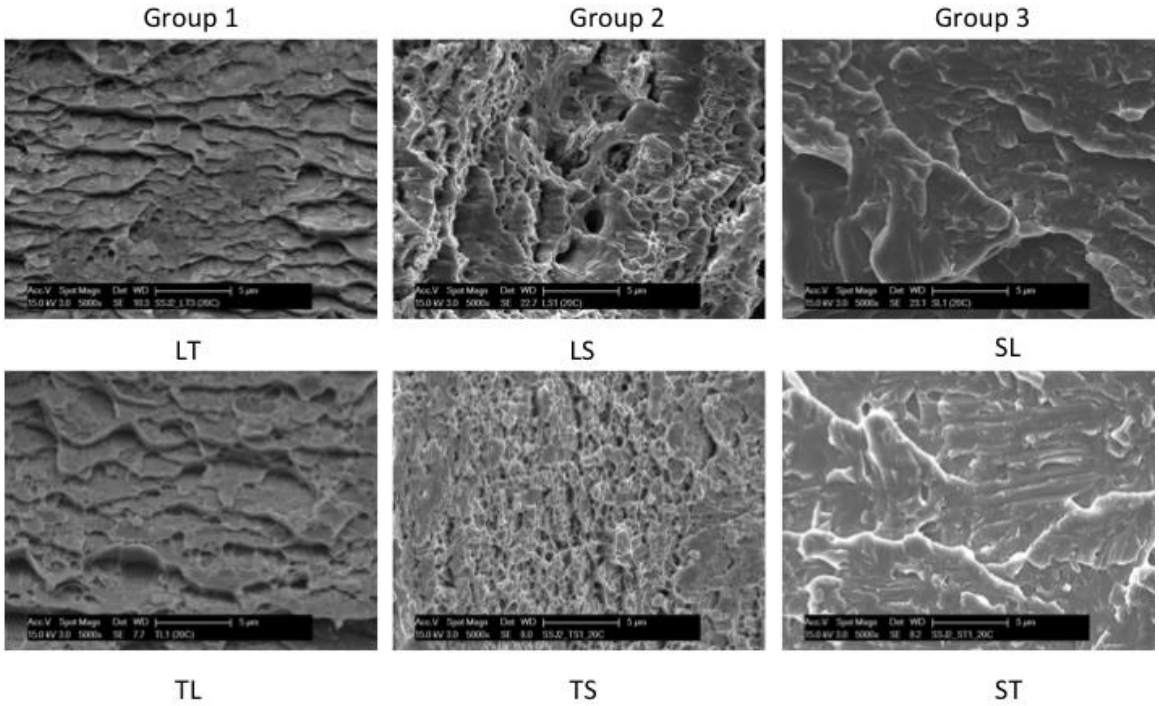


Figure 6. SEM fractographs of NFA-1 tensile specimens showing by group: (1) shear-leaps like features for LT/TL, (2) dimple like features for LS/TS, and (3) cleavage features for SL/ST orientations, respectively.

Tensile curves for tests performed at 600°C in all six orientations are shown in Fig. 7. Like room temperature tensile properties, LT oriented specimen again showed the highest strength with an average value of 584 MPa 0.2% YS and 630 MPa UTS. TL orientations again exhibited similar tensile properties as for LT, but with slightly lower strength. The LS and TS orientations (group 2) have intermediate strength (528-543 MPa 0.2%YS and 555-589 MPa UTS) with the highest total total strain (~23%). Whereas group 3 (SL and ST) specimens failed in a brittle manner with lower strength at room temperature, at 600°C the same orientations exhibited comparable strength (~ 483 MPa 0.2%YS and

~535 MPa UTS) as the other groups. As shown in Figure 8, all the orientations failed in a completely ductile manner with the total elongation as high as 13.5%. In all cases, the uniform elongation is quite low, varying between 1.1-2.3%. No delaminations were observed in any case. Since the overall strength and ductility are comparable at 600°C, an important conclusion is that NFA-1 exhibits nearly isotropic tensile behavior at higher temperature.

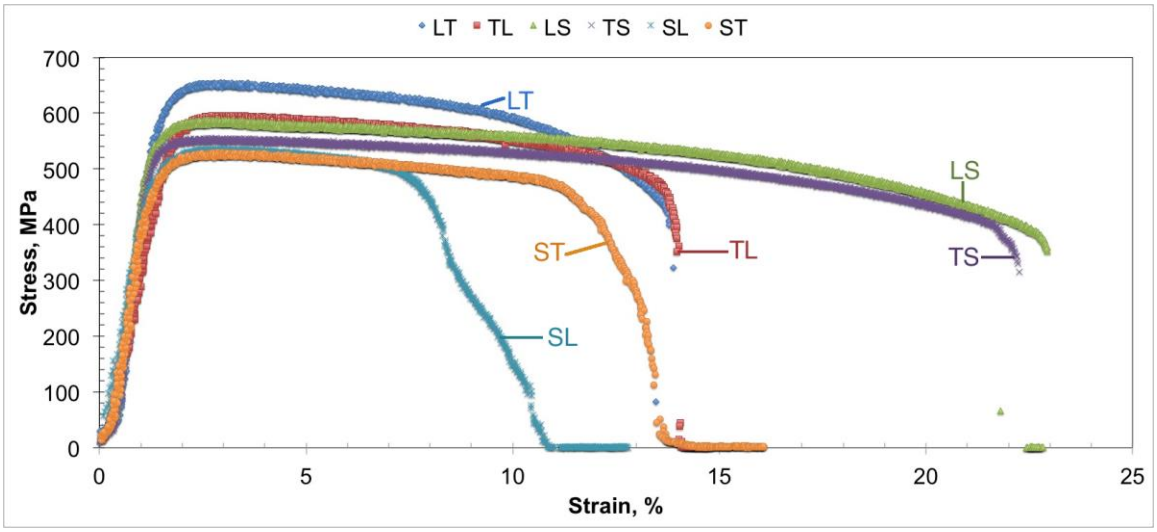


Figure 7. Engineering stress-strain curves of NFA1 tensile specimens tested at 600°C all six orientations.

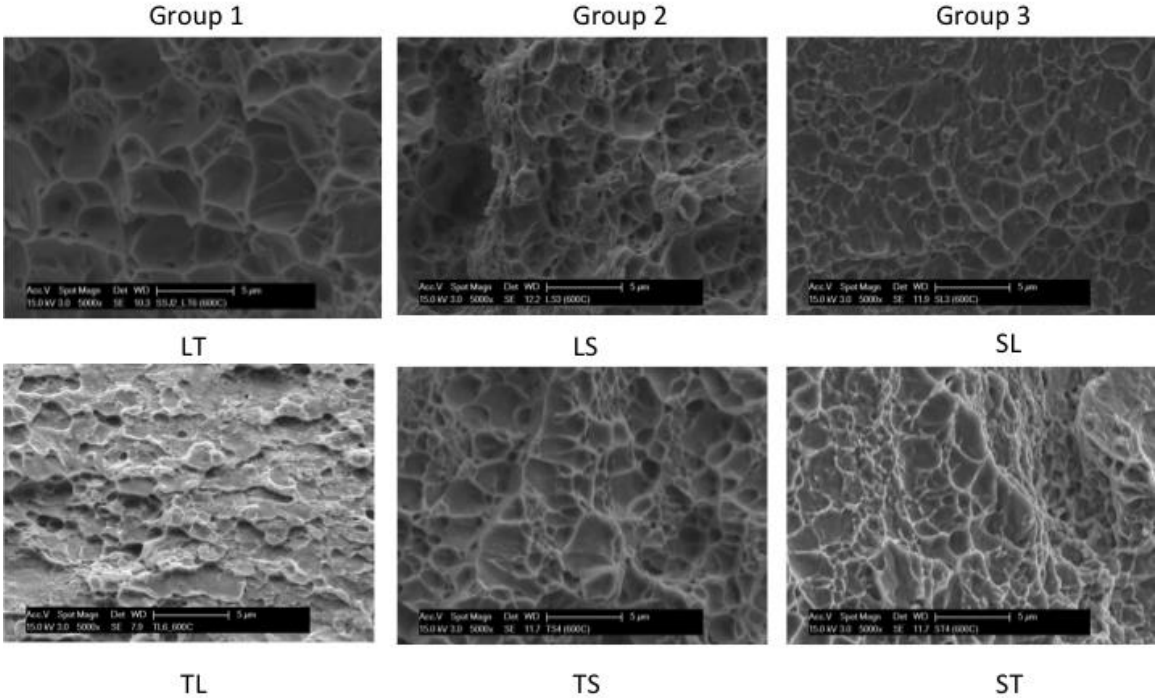
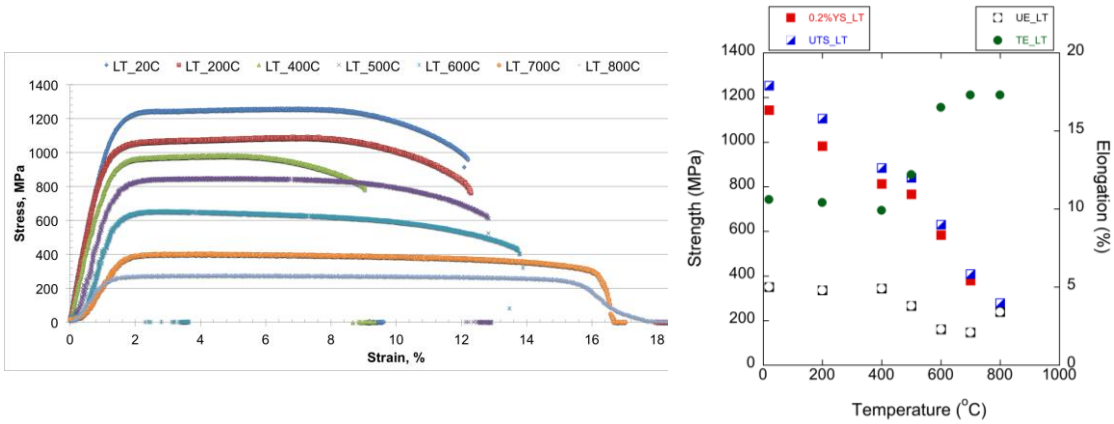


Figure 8. High magnification (x5000) SEM fractographs of NFA-1 tensile specimen fracture surface at 600°C showing dimple like features in all six orientations.

The tensile properties were characterized in the strongest (LT) and the weakest (ST) orientations from ambient temperature to 800°C and the results are shown in Figs. 9 and 10, respectively. Figure 9a is

shows the typical stress-strain curves of LT specimens as a function of temperature and Fig. 9b summarizes the various tensile properties. It can be clearly seen that the strength was decreased with increasing temperature. However, the rate of change of strength is much lower below ≈ 500°C, followed by a sharp decrease from 500°C to 800°C. This might be due to associated with a transition to viscoplastic creep even at the high strain rate of 10^{-3}/s. The strength of the NFA1 material at LT orientation is still high (>250 MPa) at 800°C. Despite the high strength, the material also exhibited relatively high total elongation (10-18%) over the entire temperature range. The uniform and total elongation were almost constant up to 400°C and then followed by a decrease in the uniform elongation up to 700°C while total elongation increased to 18% at 800°C due to the viscoplastic creep behavior.



(a) (b)

Figure 9. (a) Engineering stress-strain curves for the LT oriented NFA1 tensile specimens as a function of temperature, and (b) summary of their tensile properties.

Fig. 10a shows the tensile curves for the ST orientation. Fig. 10b plots the various corresponding tensile properties of ST oriented specimen as a function of temperature. A small amount of yielding and strain hardening is observed at 100°C that but the specimen again fails in a brittle manner. Significant strain occurred at 200°C with failure by ductile fracture. The maximum stress was similar in all three cases ranging fro 850 to 900°C. The total elongation increased almost linearly with temperature, from 0.5% at ambient temperature to 16% at 800°C, while the corresponding uniform elongation increased from 0.5% at ambient temperature to 5% at 200°C, then decreasing up to 800°C. The low temperature behavior in the ST orientation can be rationalized by a ductile to brittle transition in the alloy matric toughness that permitted cleavage cracking only between ambient temperature and 100°C.

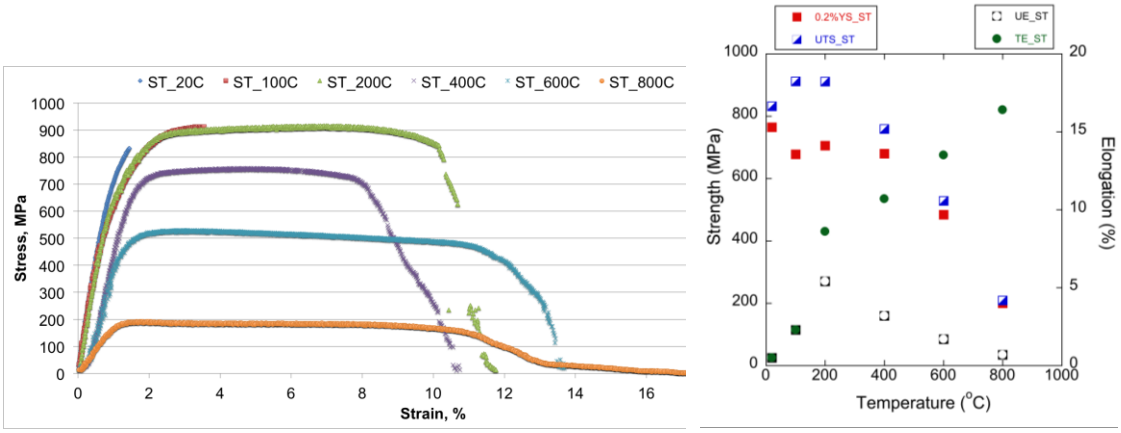
While not the topic of this report, the corresponding fracture toughness of NFA-1 is also strongly affected by the pre-existing cracks lying in planes parallel to the plat surface and normal to the thickness direction. In this case the delamination actually increase the fracture toughness. The fracture data are shown in Fig. 11 and discussed in some detail elsewhere [1]. We only briefly note that the enhanced toughness and very low ductile to brittle transition temperature In NFA-1 are due to the relaxation of high triaxial crack tip stresses upon delamination accompanied by a transition from a plane strain to a plane stress state condition.

Finally, creep rate and creep rupture time and ductility are also highly anisotropic and affected by the underlying deformation structure. However, as shown in Larson-Miller plot of the minimum creep rate shown in Figure 12, NFA-1 has a high temperature strength that is comparable to other similar alloys such as MA957.

Discussion and Summary

The tensile property of FCRD NFA1 material was fully characterized at six different orientations at varying temperature up to 800°C. In contrast to the LT orientation, NFA-1 failed in the brittle elastic region much

at a much lower stress at ambient temperature when loaded in S (short) direction. The causes of this behavior are under investigation.



(a) (b)

Figure 10. (a) Engineering stress-strain curves of ST oriented NFA1 tensile specimens as a function of temperature, and (b) summary of their tensile properties.

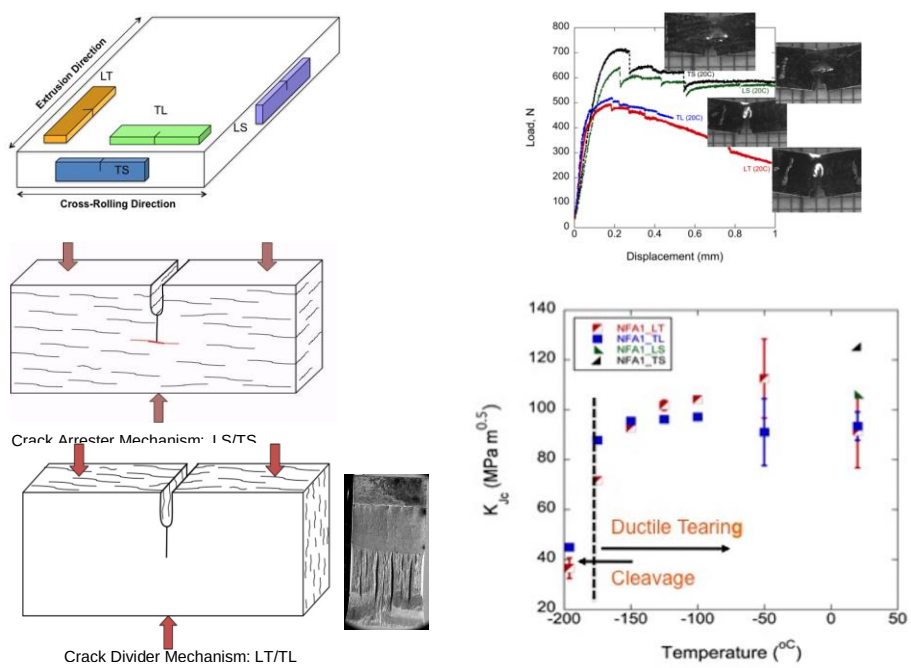


Figure 11, A pictorial summary pictorially illustrating the effect of delamination cracking on the ftactur behavior of NFA-1 as discussed in more detail elsewhere [1]

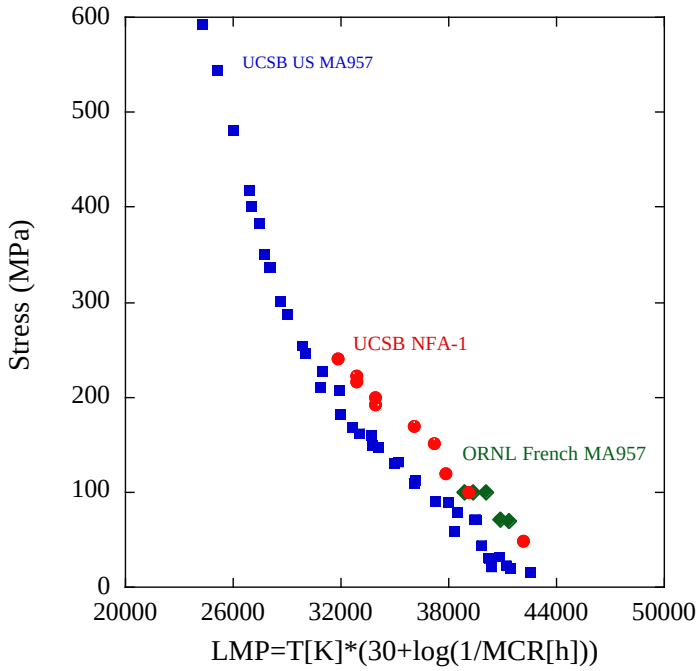


Figure 12. A comparison of the creep strength of NFA-1 with US and French heats of MA957 based on a Larson Miller minimum creep rate plot.

Acknowledgments

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References

[1] G.R. Odette, “Recent progress in developing and qualifying nanostructured ferritic alloys for advanced fission and fusion applications,” JOM. 66 (2014) 2427.
[2] N.J. Cunningham, Y. Wu, G.R. Odette, D.T. Hoelzer, S.A. Maloy, “ Characterization of a larger best practice heat of 14YWT in annealed powder, HIP consolidated and extruded forms,” DOE/ER-0313/54 (2013), DOE Fusion Reactor Materials Program Semiannual Progress Report (2013).