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Microstructural Characterization of FeCrAl C26M tubes

Fuel Cycle Research & Development

***Prepared for
U.S. Department of Energy
Advanced Fuels Campaign***

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06/29/18

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SUMMARY

The present report summarizes the initial nanoindentation characterization of a FeCrAl alloy, C26M. Microstructural analysis using electron backscatter diffraction showed a highly textured system with the axial surface favoring orientations near the [110] and the longitudinal surface near the [111]. A large spread of grain sizes was observed from a few μm 's to 100 μm with an average grain size of $\sim 70 \mu\text{m}$. Nanoindentation with a Berkovich tip measured a similar hardness and modulus of $\sim 4.4 \text{ GPa}$ and $\sim 240 \text{ GPa}$ on the two surfaces, respectively. Mounting specimens in epoxy was found to be the cause of a lower stiffness and, consequently, a lower hardness and modulus value. Spherical nanoindentation was measured on the longitudinal surface of the tube to determine the nanoindentation modulus and nanoindentation yield strength. Using a 10 μm tip, the measured modulus was $\sim 220 \text{ GPa}$ and the yield strength was $\sim 1.4 \text{ GPa}$. Indentation with a 100 μm tip resulted in a measured modulus value of $\sim 220 \text{ GPa}$ and yield strength of $\sim 1.1 \text{ GPa}$. Some early characterization of the differences in spherical nanoindentation response of the axial and longitudinal surfaces showed a small difference between the two, but a more detailed analysis with a stiffer mounting configuration is needed to draw conclusions.

TABLE OF CONTENTS

SUMMARY	iv
1. Introduction	1
2. Materials and Methods	1
3. Results and Discussion	1
3.1 Nanoindentation	1
3.2 Spherical Nanoindentation	3
4. Conclusions and Future Work	5
5. References	6

FIGURES

Figure 1 - EBSD inverse pole figure maps of the (a) axial and (b) longitudinal surface of the C26M tube, and (c) axial and (d) longitudinal surfaces of the C26M non-weld tube. Scale bar for all images is provided in (b).	2
Figure 2 - Representative nanoindentation (a) hardness and (b) modulus data for C26M and C26M non-weld specimens on the axial and longitudinal tube surfaces.	3
Figure 3. Spherical nanoindentation stress-strain curve on the longitudinal surface of the C26M tube using (a) 10 μm indenter with a (b) IPF map of the indent locations, and (c) 100 μm indenter with a (d) IPF map of the indent locations, respectively. The scale bar in (b) applies to both (b) and (d).	4

TABLES

Table 1. Chemical composition of FeCrAl C26M alloy.	1
Table 2. Summary of nanoindentation measurements. Hardness and modulus averages were made over a displacement range of 400-500 nm. Sample averages come from 25-36 tests spaced 50 μm to place indents in multiple grains. The error bars are $\pm$ one standard deviation.	3
Table 3. Summary of nanoindentation modulus and nanoindentation yield strength obtained from 10 μm and 100 μm tips.	4
Table 4. Summary of nanoindentation modulus and nanoindentation yield strength obtained from 10 μm and 100 μm tips on the axial and longitudinal surface of C26M tubing mounted in epoxy.	5

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1. Introduction

Among several candidates for “accident tolerant” fuel cladding, FeCrAl alloys are among the top choices due to their superior high temperature oxidation resistance, aqueous corrosion resistance, low radiation-induced swelling, and tolerance to loss-of-coolant accident conditions [1-4]. It is important to note that despite their higher neutron absorption cross-section compared to zirconium-based alloys, their mechanical and chemical stability over a range of environment make this alloy and attractive candidate to others (i.e. SiC-based cladding). The ongoing work led by Oak Ridge National Lab has targeted an optimized FeCrAl alloy for tube processing and implementation in light water reactors [5]. Recent testing and evaluation has led to a down selected alloy, C26M. At present, very little microscale mechanical testing has been performed on this alloy. As part of a wider effort to evaluate the mechanical properties of this alloy, we perform initial microstructural characterization and testing using nanoindentation techniques.

2. Materials and Methods

Two samples of a down-selected FeCrAl alloy (C26M) were provided in tube form with a composition given in Table 1. One tube (Heat #17025001), was sectioned to expose both the axial surface of the tube and the longitudinal surface of the tube and referred to simply as C26M. Specimens were taken from another tube where tungsten inert gas (TIG) welding was performed to join tubes. Initial characterization of the tube was performed far from the weld region (referred to as C26M non-weld) while the weld area is undergoing separate characterization and will be in a future report. All specimens were ground using successive SiC grit papers down to a grit of 1200. Samples were then polished first using a 0.25 μm diamond solution and finished with a 0.04 μm silica solution.

Table 1. Chemical composition of FeCrAl C26M alloy.

Alloy ID	Fe	Cr	Al	Y	Mo	Si	Nb	C	S	O	N
C26M	bal	11.87	6.22	0.030	1.98	0.2	-	<0.01	0.005	-	-

Scanning electron microscopy of the polished tubes was performed in a FEI Inspect equipped with an EDAX system for acquiring electron backscatter diffraction patterns (EBSD), and obtained with an electron energy of 20 keV. Nanoindentation tests were performed on a Keysight G200 Nanoindenter with a diamond, pyramidal (Berkovich) tip to a final displacement of 1,000 nm with a constant strain rate (loading rate divided by the load) of 0.05 s^{-1} . Continuous stiffness measurements (CSM) were performed at a frequency of 45 Hz and 2 nm displacement amplitude. Hardness and modulus measurements were determined using the Oliver-Pharr method [6]. The tip area function was calibrated by indenting fused silica and using tip properties with a Young’s modulus and Poisson’s ratio of 1130 GPa and 0.07 (diamond). Spherical nanoinindentation tests were performed using two tips with 10 μm and 100 μm radii, respectively. Nanoindentation was performed to a depth of 500 nm using similar system parameters to the nanoindentation with a Berkovich tip.

3. Results and Discussion

3.1 Nanoindentation

Figure 1a and b provide inverse pole figure (IPF) maps of the C26M tube along the axial and longitudinal surfaces of the specimen, respectively. Fig. 1c and d provide maps of the region far from the weld of the axial and longitudinal surfaces of the specimen, respectively. The axial surface shows a

strong texture to the [110] direction and the longitudinal surface shows texturing along the [111] direction. In both specimens, the grains do not display a high degree of anisotropy (equiaxed) on either surface. Small grains of a few μm 's and large grains on the order of 100 μm were observed, with an average grain size $\sim 70 \mu\text{m}$.

Fig. 2 provides a comparison of the nanoindentation hardness and modulus of the four specimens prepared. All samples exhibited a hardness between 4-4.5 GPa and a modulus of 220-240 GPa. Table 2 provides a summary of the nanoindentation data. There are, however, notable differences between each specimen. C26M non-weld specimens were mounted on an aluminum stub with a small amount of super glue to bond the specimen to the stub. C26M specimens were encased in epoxy mixed with nickel powder. The large difference in modulus and hardness between these sets (at low displacements) arises from the compliance of the respective supporting media. However, beyond differences in the mounting configuration, there is very little difference in hardness between the two surfaces of either tube specimen.

Figure 1 - EBSD inverse pole figure maps of the (a) axial and (b) longitudinal surface of the C26M tube, and (c) axial and (d) longitudinal surfaces of the C26M non-weld tube. Scale bar for all images is provided in (b).

Figure 2 - Representative nanoindentation (a) hardness and (b) modulus data for C26M and C26M non-weld specimens on the axial and longitudinal tube surfaces.

Table 2. Summary of nanoindentation measurements. Hardness and modulus averages were made over a displacement range of 400-500 nm. Sample averages come from 25-36 tests spaced 50 μm to place indents in multiple grains. The error bars denote one standard deviation.

Sample	Hardness (GPa)	Modulus (GPa)
C26M Non-weld Axial	4.43 ± 0.12	236.5 ± 7.11
C26M Non-weld Longitudinal	4.48 ± 0.14	249.2 ± 7.05
C26M Tube Axial	4.13 ± 0.11	227.4 ± 5.94
C26M Tube Longitudinal	4.01 ± 0.14	218.0 ± 7.93

3.2 Spherical Nanoindentation

Nanoindentation using spherical tips imparts higher loads to a sample than a Berkovich tip at similar depths. For this type of indentation, a stiff mounting configuration is needed. From the results obtained from nanoindentation with a Berkovich tip, samples mounted on an aluminum stub with a thin layer of superglue provide the highest stiffness of those tested. The longitudinal surface of the C26M tube was selected for detailed spherical nanoindentation analysis.

Fig. 3 provides the calculated stress-strain values using a process outlined elsewhere [7]. Nanoindentation stress-strain curves do not have a large amount of scatter. We note that the grain orientations where indents were placed are all similar (i.e. near the [110]). This is not to say that no orientation-dependence is expected, as the number of available slip systems largely determines plasticity behavior (i.e. yield strength, strain hardening), but that similar orientations result in similar slip systems being activated. It is also worth noting that the choice of sample preparation did not result in any pop-ins in the stress-strain curves as mechanical polishing (e.g. vibratory polishing) introduces a “balanced” density of defects so as to avoid significantly increasing the yield strength [8].

Table 3 provides a summary of the calculated nanoindentation moduli and nanoindentation yield strengths. The calculated nanoindentation moduli of the sample from indents using a tip with a radius of 10 μm and 100 μm are 217 GPa and 219 GPa, respectively. The yield strength from a 10 μm indent was found to be ~1 GPa while that from a 100 μm indent was ~1.3 GPa. We note the difference between the modulus calculated from spherical indentation, which agrees closely with the modulus of iron, and that from Berkovich indentation differs by a small, but significant amount. It is important to note that the elastic properties of the material are measured during the loading portion with a spherical tip. In turn, this

more accurately captures the initial microstructure of the sample. With Berkovich indentation, the modulus is measured upon unloading the tip, thus measuring elastic properties after significant plastic deformation.

Figure 3. Spherical nanoindentation stress-strain curve on the longitudinal surface of the C26M tube using (a) 10 μm indenter with a (b) IPF map of the indenter locations, and (c) 100 μm indenter with a (d) IPF map of the indenter locations, respectively. The scale bar in (b) applies to both (b) and (d).

Table 3. Summary of nanoindentation modulus and nanoindentation yield strength obtained from 10 μm and 100 μm tips.

C26M	10 μm		100 μm	
	Modulus (GPa)	Yield Strength (GPa)	Modulus (GPa)	Yield Strength (MPa)
	219.4 ± 0.173	1.37 ± 0.08	217.1 ± 3.7	1.02 ± 0.05

We note that the nanoindentation yield strength appears nearly a factor of 2 larger than that measured from bulk uniaxial tensile tests. For spherical nanoindentation, the mixed stress state does not permit a direct comparison to results obtained from tests with a uniaxial stress state (i.e. tension, compression). Finite element simulations on an isotropic material have shown that the uniaxial and nanoindentation stress states can be related by [9],

$$\sigma = \frac{\sigma_{ind}}{2.2}$$

Where σ_{ind} is the nanoindentation stress state and σ is the uniaxial stress state. Applying this constant factor to the values in Table 3 now more closely match the expected yield values in bulk tensile tests.

Although more detailed testing was performed strictly on the longitudinal surface, some early spherical nanoindentation was performed on the axial surface of the tube. In order to obtain a good surface finish, both parts of the tube were mounted in epoxy. Table 4 summarizes the results. It is worth noting that despite the influence of the soft mount and a large deviation in the data, nanoindentation with both tip sizes suggest that the longitudinal surface possesses lower strength than the axial direction. More detailed examination via indentation with a stiffer mounting configuration is needed to verify the observations.

Table 4. Summary of nanoindentation modulus and nanoindentation yield strength obtained from 10 μm and 100 μm tips on the axial and longitudinal surface of C26M tubing mounted in epoxy.

C26M		10 μm		100 μm	
		Axial	Longitudinal	Axial	Longitudinal
	Modulus (GPa)	205.1 ± 4.68	207.6 ± 6.90	197.0 ± 1.41	196.6 ± 1.72
	Yield Strength (GPa)	1.23 ± 0.17	1.12 ± 0.26	1.25 ± 0.05	1.20 ± 0.1

4. Conclusions and Future Work

In this study, we have performed an initial characterization of the microstructure of C26M tubing of two different heats. Nanoindentation with a Berkovich tip was utilized to evaluate the hardness of the axial and longitudinal surfaces of the two tubes. The results indicate that the hardness on either surface are similar. The biggest difference between the two sets arose from the mounting configuration. Adhering samples to an aluminum stub with a thin layer of adhesive provided the stiffest configuration.

Spherical nanoindentation on the longitudinal surface of the C26M tube provided additional insights into the indentation response of the alloys. Nanoindentation with spherical tips of 10 μm and 100 μm radius were performed and a measured modulus of between 217-219 GPa was measured, in better agreement with Young's modulus for iron. Nanoindentation yield strengths were measured to be ~ 1 GPa and ~ 1.4 GPa for the 10 μm and 100 μm tips, respectively.

Future work for characterizing the system will shift focus from nanoindentation (high throughput) towards mechanical testing techniques that offer high quality information:

- Microscale shear specimen designs were recently developed and tested on Cu systems (single crystal and nanocrystalline). Initial work has begun on C26M in an effort to characterize the oft overlooked shear properties.
- A high throughput method for fabricating microscale pillars and cantilevers is nearing completion. This technique will enable fabrication of dozens of pillars and cantilevers in less than an order-of-magnitude time that is required with traditional FIB preparation.
- The development of nanoindentation techniques to characterize the ductile-to-brittle transition temperature will be performed. Beyond using cantilevers measure changes in fracture toughness, nanoindentation provides one of the only feasible options for measuring the DBTT of materials after heavy ion irradiation.

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