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Shear Punch Testing of BOR60 Irradiated HT-9 and 14YWT

Fuel Cycle Research & Development

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SUMMARY

Ferritic/martensitic (FM) steels and nanostructured ferritic alloys (NFA) are being developed for next generation reactor applications. Applications such as transmutation of long lived isotopes in used fuels require cladding materials that can withstand high irradiation doses, potentially in the hundreds of displacements per atom (dpa). The FM steels in this report include HT-9 steel, which is a Fe-12Cr steel with additions of Mo, Mn, Ni, V and W. An oxide dispersion strengthened (ODS) 14YWT is also presented, which has a distribution of nano-scale oxide particles throughout the microstructure. Shear punch testing was performed on both steels. The material was neutron irradiated at the BOR60 reactor in Russia and transferred to LANL for shear punch mechanical testing. These results are complementary to a previous report, also from a BOR60 irradiation [1].

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1. Introduction

Cladding materials for Generation IV fast reactors, such as the sodium fast reactor, will be subjected to irradiation damage levels in the hundreds of dpa and temperatures of 400-600 °C, which is above those required for current generation light water reactors. Doses expected at lower temperatures (e.g. 350-400C) are lower but strong hardening is observed at those temperatures. Thus data is needed to characterize mechanical properties after low temperature irradiation also.

Use of ferritic/martensitic steels such as HT-9 or oxide-dispersion strengthened ferritic steels such as 14YWT are candidate materials to meet these service conditions based on their resistance to swelling, high thermal conductivity, and sufficient high temperature mechanical properties. It is necessary, however, to determine the changes to mechanical properties after neutron irradiations at conditions expected for service of the cladding materials. Significant hardening is observed in HT-9 with irradiation while less hardening is observed for the ODS steels. ODS steels are produced such that a distribution of nano-scale oxide precipitates are in the microstructure. These nano-oxide precipitates not only strengthen the material, but also may act as sinks, or recombination sites for irradiation induced interstitial and vacancy defects. In addition, ductility (in terms of work hardening strain) is known to decrease after low temperature irradiation.

2. Materials and Methods

2.1 Materials

Two alloys, HT-9 and 14YWT, were neutron irradiated and in the form of 3 mm discs and thicknesses around 0.25 mm. Compositions are given in Table 1 for both alloys. The HT-9 was from the samew lot as the AC03 duct. The 14YWT material was from the SM13 lot.

Table 1: Compositions of alloys.

Alloy ID	Fe	Cr	Y	W	Ti	O	C	Si	Mn	Ni	Mo	V
14YWT	bal	15.9	0.03	0.52	0.07	0.04	0.03	0.07	-	-	-	-
HT-9	bal	12.49	-	0.52	-	0.002	0.201	0.28	0.41	0.60	1.07	0.29

2.2 Shear punch testing

Shear punch testing was carried out with a fixture seen in Figure 1 and 2. TEM specimens (3 mm diameter, between 0.18 and 0.26 mm thick) were subjected to a 1 mm punch in uniaxial compression. The punching is carried out on an Instron 30 kN screw driven load frame located in the hot cells in Wing 9 of the CMR building at LANL. Displacement rate was 0.127 mm/min. The raw Load-Displacement data is transformed into an Effective Shear Stress-Displacement curve via simple calculations. The Effective Shear Stress (ESS) is the *Load/Area*, with *Area* being the deformed region, which is the *circumference of the punch* divided by the *thickness of the TEM sample*. This ESS vs Extension curve can be correlated to tensile yield and UTS via simple relationships, more details can be seen in references 2-4. The thickness of each TEM sample is measured before testing with a micrometer in the hot cells. Due to the sample fabrication methods, punching from a sheet of material, and subsequent cupping of the samples, as well as some burrs on the sample (Figure 3) there was some uncertainty in the initial thickness measurements of each sample. A new micrometer with 1/16” ball tip was fabricated to measure the sample’s thickness. This thickness measurement, along with comparison to measurements on the sheets of raw material before punching, were used to correct some of the early tests.

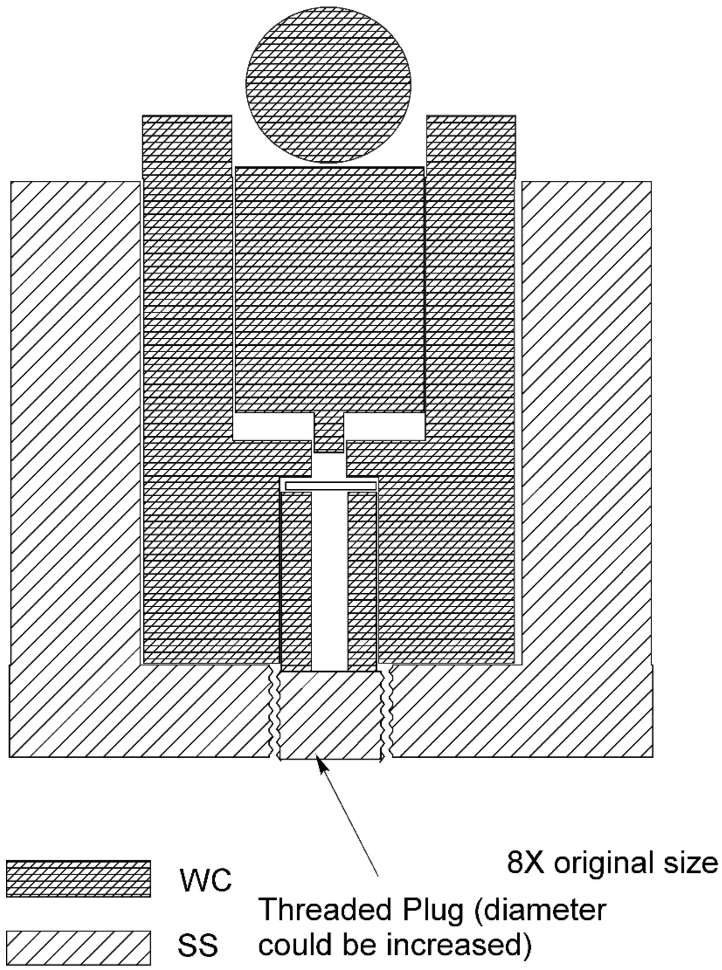

Figure 1: Schematic of Shear Punch Test fixture for 3 mm x 0.25 mm TEM samples using a 1 mm punch.

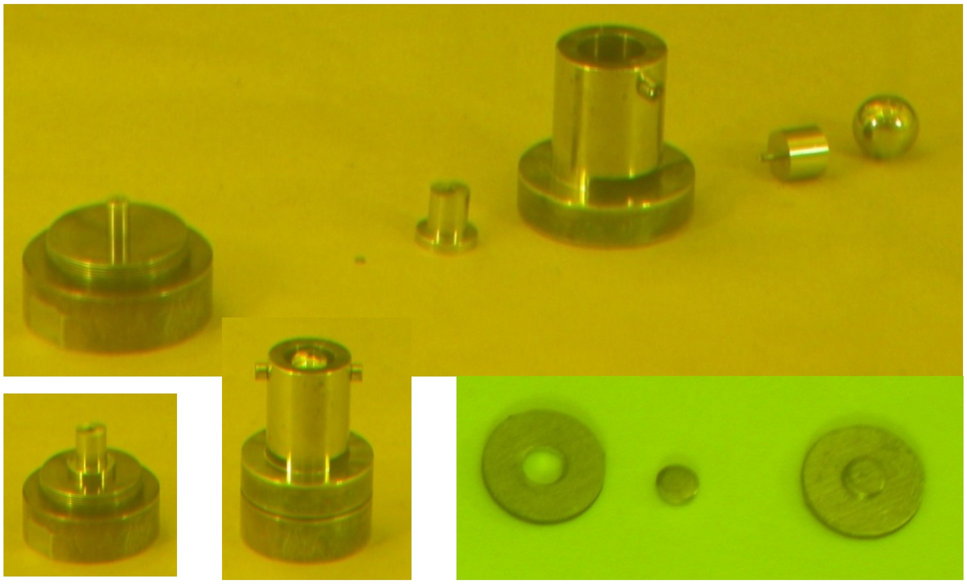

Figure 2: Images of Shear Punch Test fixture for 3 mm x 0.25 mm TEM samples using a 1 mm punch.

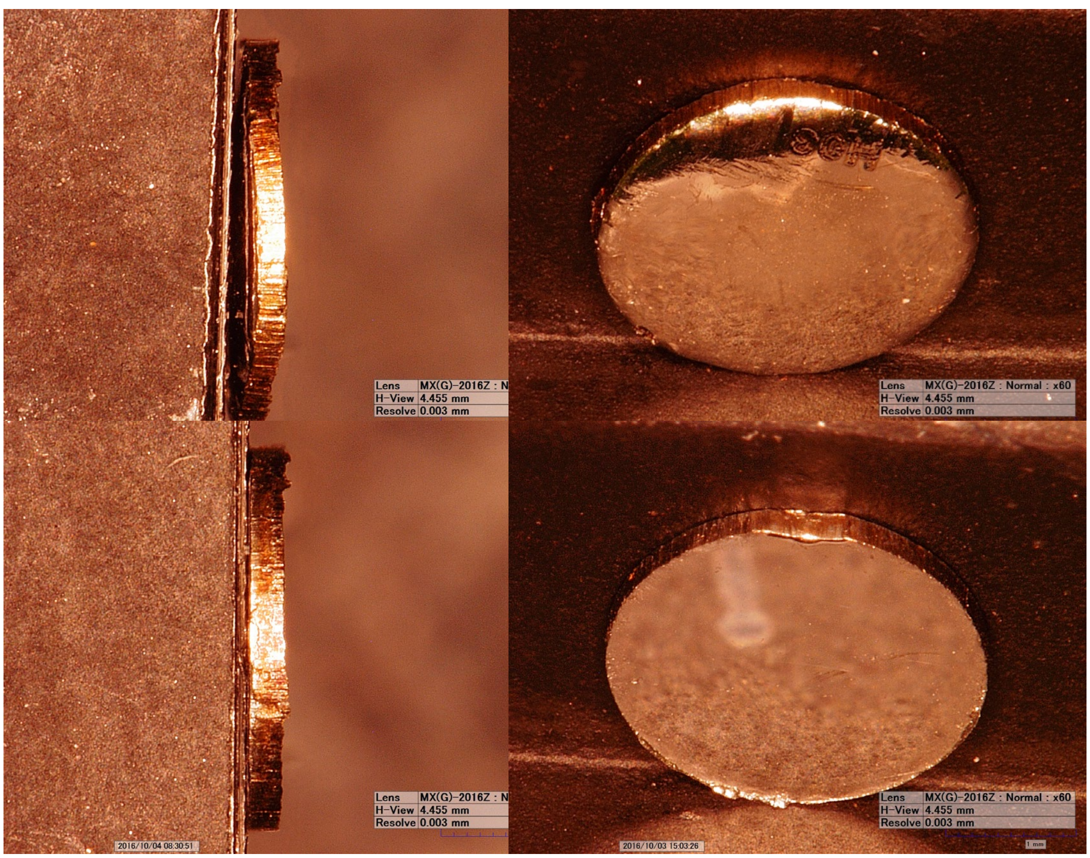

Figure 3: TEM samples showing cupping (top) and burrs (bottom) due to punching from sheets.

3. Results and Discussion

3.1. Mechanical properties

Shear punch tests were conducted on both HT-9 and 14YWT (ODS) alloys at irradiation conditions described in Table 2. Control materials that were not irradiated were also tested for comparison. In Table 2, comparisons can be made between the two alloys and irradiation conditions in terms of yield behavior, work hardening and ductility. The 1% offset effective shear stress is determined at 1% strain past the linear region of the plots, where strain is calculated by dividing displacement by sample thickness. Work hardening strain was calculated as strain between the 1% offset effective shear stress and maximum effective shear stress, with strain calculated by dividing displacement by sample thickness. Variations in the sample thicknesses are shown in Table 2 and are significant, particularly for the HT-9 samples. Work hardening $\Delta\sigma$ is the difference between the 1% offset effective shear stress and maximum effective shear stress.

From Table 2, it is shown that irradiation temperature has an impact on both alloys mechanical response, such that at the lower irradiation temperatures, 378 and 425 °C, 1% offset effective shear stress and maximum shear stress is highest compared to the higher irradiation temperatures, 460 and 524 °C. Compared to unirradiated control samples the HT-9 irradiated at 524 °C had comparable 1% offset effective shear stress and maximum effective shear stress values. In the case of 14YWT irradiated at 524 °C compared to unirradiated control samples, slight hardening was observed shown as an increased 1% offset effective shear stress and maximum effective shear stress.

Work hardening occurred in both alloys after yielding with HT-9 generally exhibiting larger work hardening strain than the ODS alloy. Increase in effective shear stress work hardening $\Delta\sigma$ was generally higher for the 14YWT material than HT-9, however, the samples irradiated at 524 °C showed the opposite.

Effect of dose was also investigated for the HT-9 alloy with irradiations at 17.1 dpa at 376 °C and 35.1 at 378 °C. With increase in dose, 1% offset effective shear stress increased, work hardening strain decreased, and maximum effective shear stress did not significantly change. Results are present in Table 2 and also plotted later in this report.

Table 2: Sample identifications, doses and temperature of irradiation at BOR60 reactor in Russia.

Capsule	Material	ID	Sample Thickness (mm)	Dose (dpa)	Temp (°C)	1% Offset Stress (MPa)	Max Effective Shear Stress (MPa)	Work Hardening Strain	Work Hardening Δσ (MPa)
P028	HT-9	C54	.207	35.1	378	554	733	0.29	179
		C55	.178	35.1	378	563	730	0.35	167
	ODS	Y09	-	35.1	378	-	-	-	-
		Y10	.260	35.1	378	759	1021	0.24	262
P035	HT-9	C84	.225	19.6	425	502	683	0.39	181
		C86	.260	19.6	425	540	691	0.37	151
	ODS	Y74	.250	19.6	425	809	1041	0.20	232
		Y76	.250	19.6	425	763	981	0.33	218
P037	HT-9	C47	.222	14.6	460	358	594	0.44	236
		C67	.182	14.6	460	424	583	0.40	159
	ODS	Y42	.260	14.6	460	750	1012	0.30	262
		Y47	.250	14.6	460	761	1016	0.33	255
P042	HT-9	C69	.180	15.4	524	242	588	0.52	346
		C66	-	15.4	524	-	-	-	-
	ODS	Y38	.250	15.4	524	806	1018	0.27	212
		Y45	.258	15.4	524	716	942	0.36	226
Control	HT-9	1	.249	0	RT	410	578	0.36	168
		2	.245	0	RT	358	554	0.36	196
	ODS	1	.231	0	RT	643	893	0.40	249
		2	.231	0	RT	679	913	0.35	234
P027	HT-9	C03	.230	17.1	376	520	726	0.40	206
		C00	.229	17.1	376	510	717	0.39	207
P033		C61	.241	18.6	415	480	669	0.36	189
		C23-C	.241	18.6	415	440	631	0.35	191
		C23-C2	.229	18.6	415	460	664	0.37	204
		C23	.263	18.6	415	420	579	0.31	159

Figure 5 shows isolating the effect of (a) dose at around 377 °C and (b) irradiation temperature at doses between 14.6 and 19.6 dpa. What is shown in Figure 5(a) is that between doses of 17.1 and 35.1 dpa: 1% offset effective shear stress increases with dose, maximum effective shear stress remains similar with dose, and ductility decreases with dose. Quantitative differences can be determined from Table 2. The effect of temperature, shown in Figure 5(b), generally shows that in the dose range between 14.6 and 19.6 dpa, 1% offset effective shear stress and maximum effective shear stress increases with decreasing irradiation temperature. There are some exceptions to the trend with temperature, for example, the samples irradiated at 415 °C compared to 425 °C show the 1% offset effective shear stress and maximum effective shear stress are notable higher for the 425 °C irradiation samples. Figure 5 shows the trend in 1% offset effective shear stress and maximum effective shear stress as a function of temperature for both (a) HT-9 and (b) 14YWT.

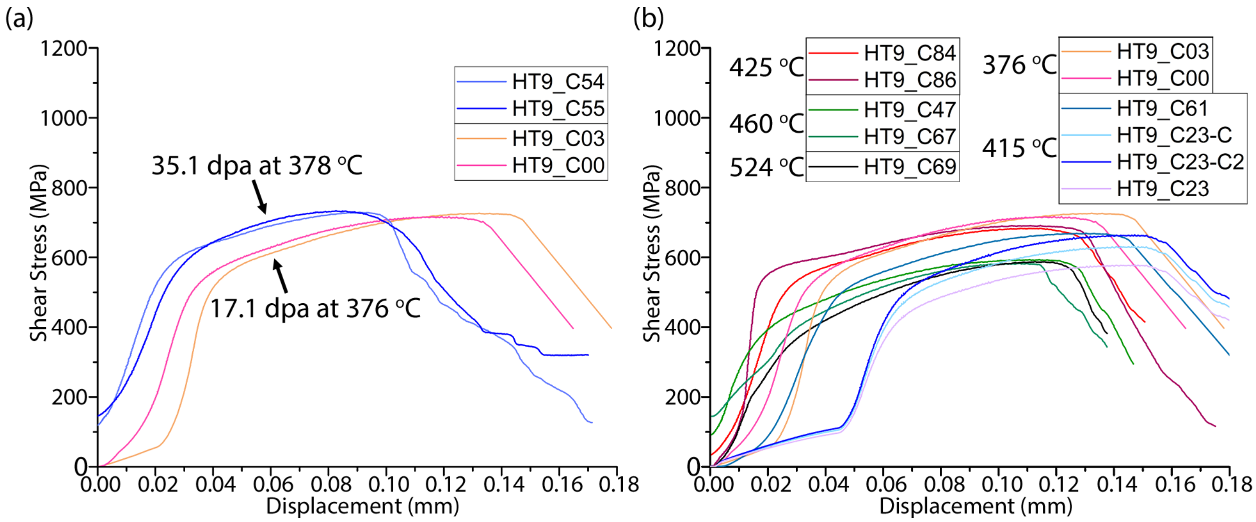

Figure 4: Comparison by isolating the effect of (a) dose at around 377 °C and (b) irradiation temperature at doses between 14.6 and 19.6 dpa. Effective shear stress vs displacement for neutron irradiated HT-9 from shear punch tests. Displacement was corrected to account for compliance in the testing system.

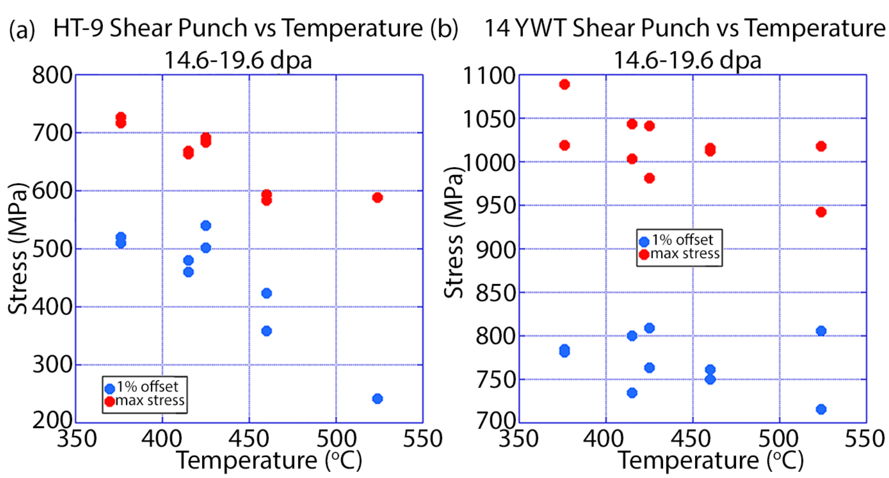

Figure 5: Comparison of 1% offset effective shear stress and maximum effective shear stress for (a) HT-9 and (b) 14YWT.

Shear punch results are plotted below in Figures 6-8 as effective shear stress vs displacement. The displacement was corrected for compliance in the test system. Shear punch shear stress vs displacement of neutron irradiated HT-9 samples are present in Figures 6-7 and neutron irradiated 14YWT samples are presented in Figure 8.

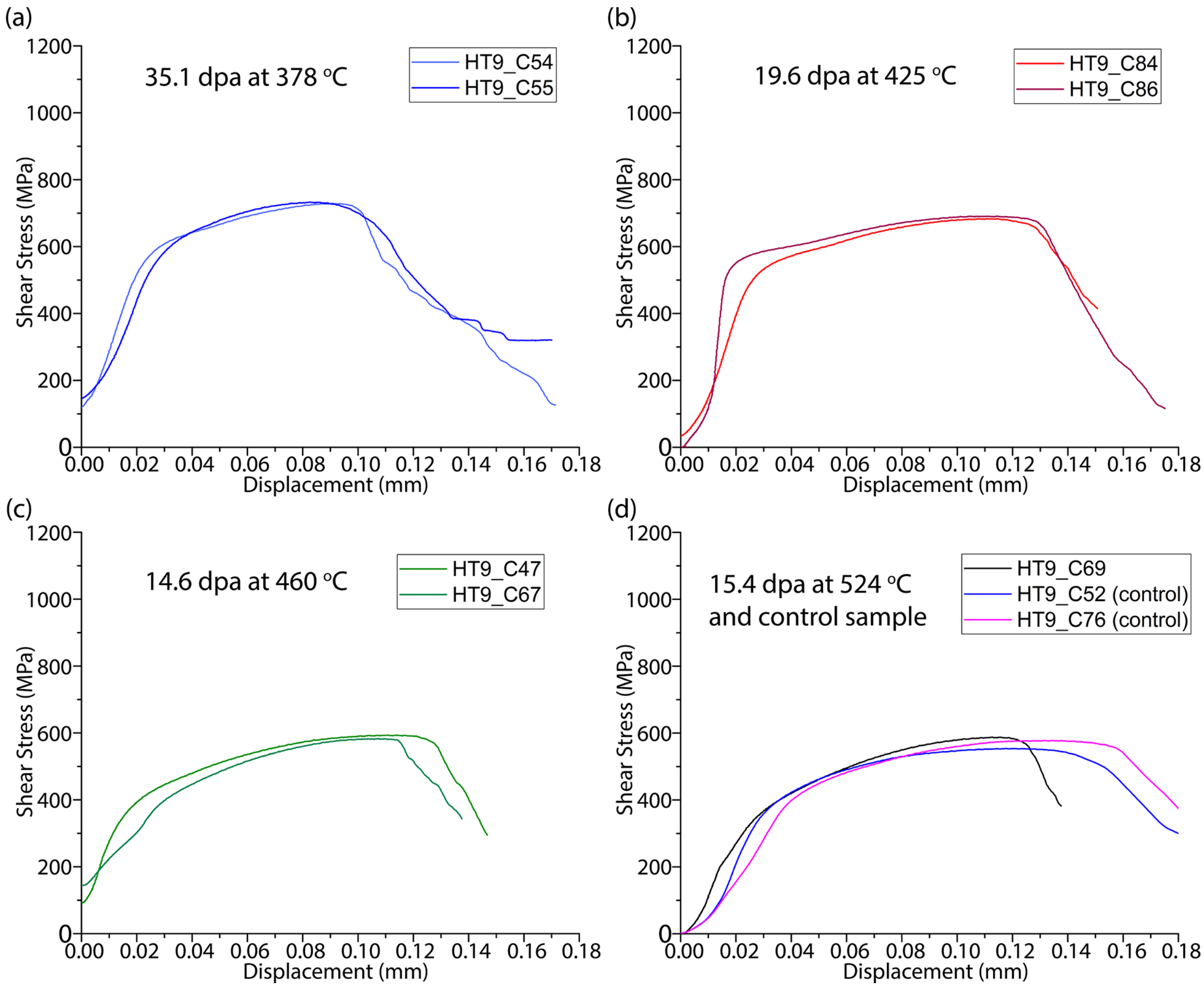

Figure 6: Effective shear stress vs displacement for neutron irradiated HT-9 from shear punch tests. Displacement was corrected to account for compliance in the testing system.

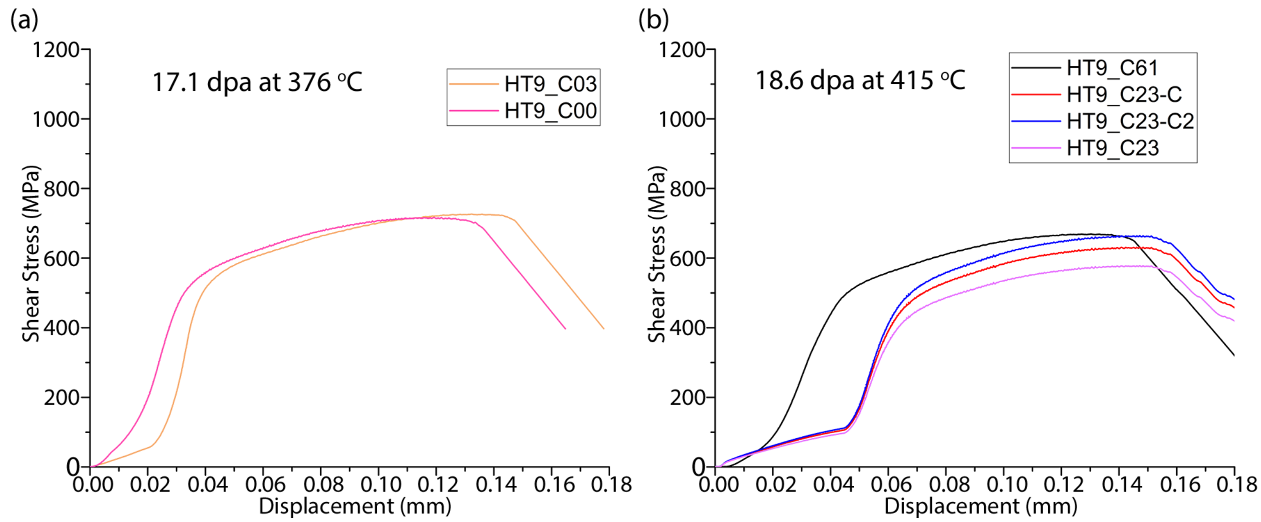

Figure 7: Effective shear stress vs displacement for neutron irradiated HT-9 from shear punch tests. Displacement was corrected to account for compliance in the testing system.

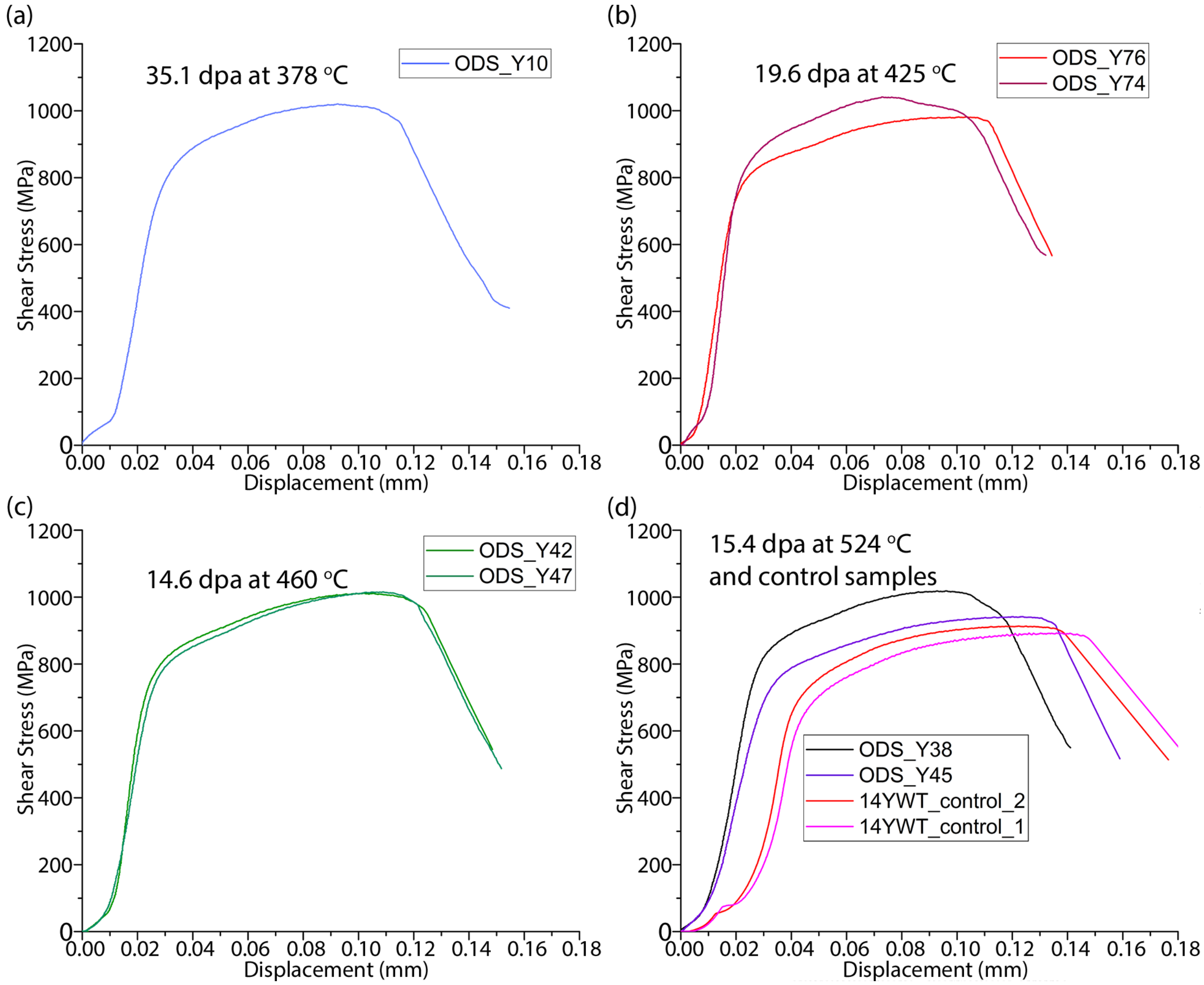

Figure 8: Effective shear stress vs displacement for neutron irradiated 14YWT from shear punch tests. Displacement was corrected to account for compliance in the testing system.

4. Conclusions

Mechanical property evolution of HT-9 and 14YWT alloys with neutron irradiation at doses between 14.6 to 35.1 dpa, and at temperatures between 376 and 524 °C are shown through shear punch testing. Response is material dependent.

HT9:

- 1% offset and maximum effective shear stress increase with decreasing temperature. Such that at the two highest irradiations temperatures, 460 and 524 °C, the mechanical response was similar to the control material.
- Increasing dose, at 376 and 378 °C, from 17.1 to 35.1 dpa increases the 1% offset effective shear stress and decreases displacement. However, the maximum effective shear stress is similar between the two doses.

14YWT:

- 14YWT exhibited less impact of irradiation on mechanical response compared to HT9. The results of tests performed on the samples irradiated at the various irradiation temperatures showed similar responses which is evidence of the improved radiation tolerance of this ODS alloy.

5. References

[1] Tarik A. Saleh, Matthew E. Quintana, Tobias J. Romero "Shear Punch Testing of BOR-60 Irradiated TEM Specimens" United States. doi:10.2172/1363742. https://www.osti.gov/servlets/purl/1363742.
[2] M.B. Toloczko, R.J. Kurtz , A. Hasegawa , K. Abe "Shear punch tests performed using a new low compliance test fixture" Journal of Nuclear Materials 307–311 (2002) 1619–1623
[3] H.T. Bach, O. Anderoglu, T.A. Saleh, et. al. Proton irradiation damage of an annealed Alloy 718 beam window Journal of Nuclear Materials 459 (2015) 103–113
[4] S.A. Maloy, T.J. Romero, P. Hosemann, M.B. Toloczko, Y. Da, Shear punch testing of candidate reactor materials after irradiation in fast reactors and spallation environments, Journal of Nuclear Materials Volume 417, Issues 1–3, 1 October 2011, Pages 1005–1008