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Shear Punch Testing of BOR-60 Irradiated TEM Specimens

Nuclear Technology Research and Development

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SUMMARY

As a part of the project “High Fidelity Ion Beam Simulation of High Dose Neutron Irradiation” an Integrated Research Program (IRP) project from the U.S. Department of Energy, Nuclear Energy University Programs (NEUP), TEM geometry samples of ferritic cladding alloys, Ni based super alloys and model alloys were irradiated in the BOR-60 reactor to ~16 dpa at ~370°C and ~400°C. Samples were sent to Los Alamos National Laboratory and subjected to shear punch testing. This report presents the results from this testing.

1. INTRODUCTION

As a part of the project "High Fidelity Ion Beam Simulation of High Dose Neutron Irradiation" an Integrated Research Program (IRP) project from the U.S. Department of Energy, Nuclear Energy University Programs (NEUP), two capsules of TEM geometry (3mm diameter by 0.25 mm thick) samples of various cladding and model alloys irradiated in the BOR-60 reactor to ~16 dpa at ~370°C and ~400°C. Dose and temperatures are reported to the current best models, more precise estimates of dose and temperature are ongoing at Terrapower. A listing of samples tested is shown in Table 1. Sample capsules were sent from the BOR-60 reactor in Dimitrovgrad, Russia to Los Alamos National Laboratory and subjected to shear punch testing. This report presents the results from this testing.

2. EXPERIMENTAL

Shear punch testing was carried out with a fixture seen in Figure 1 and 2. TEM specimens (3mm diameter, 0.25mm thick) were subjected to a 1mm punch in uniaxial compression. The punching is carried out on an Instron 30kN screw driven load frame located in the hot cells in Wing 9 of the CMR building at LANL. The raw Load-Extension data is transformed into an Effective Shear Stress-Extension 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 1-3. 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 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.

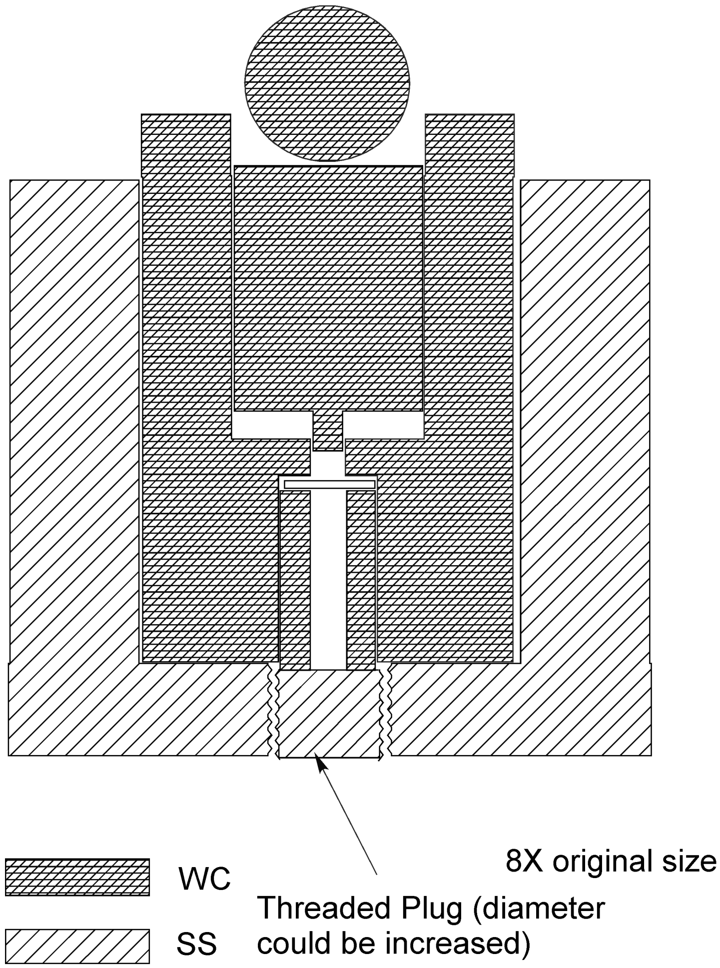

Figure 1. Schematic of Shear Punch Test fixture for 3mm x 0.25mm TEM samples using a 1mm punch.

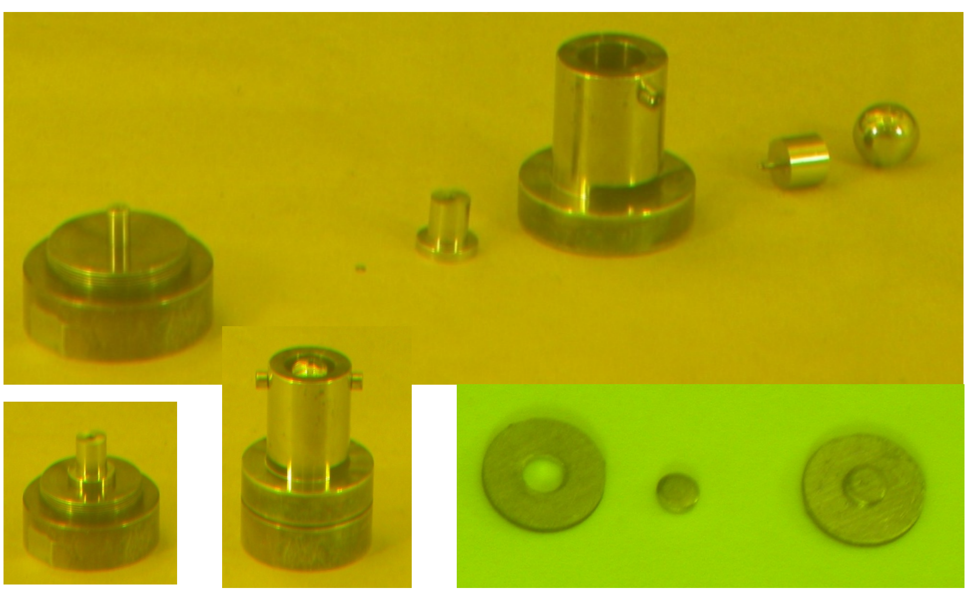

Figure 2. Images of Shear Punch Test fixture for 3mm x 0.25mm TEM samples using a 1mm punch.

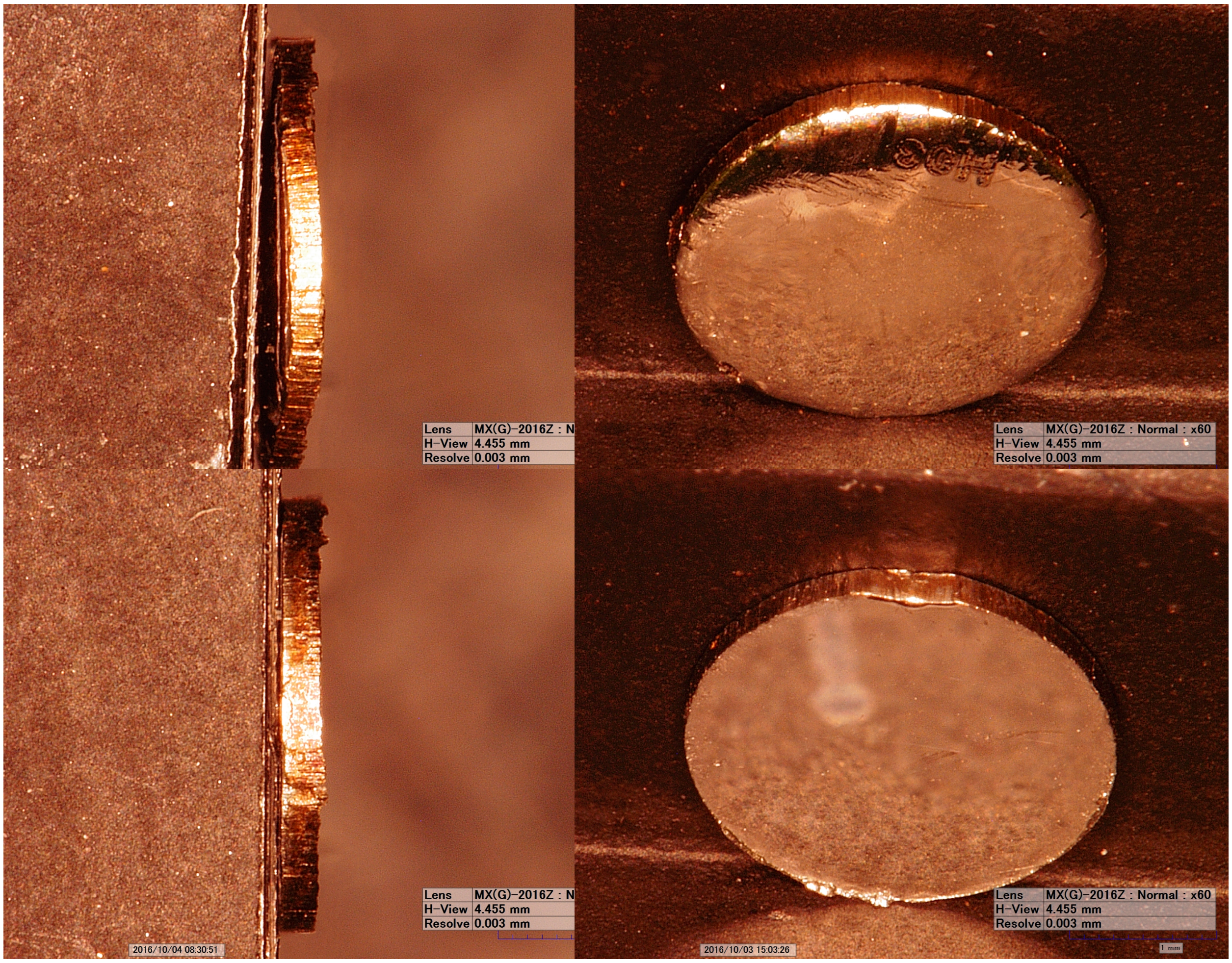

Figure 3. TEM samples showing cupping (top) and burrs (bottom) due to punching from sheets.

3. RESULTS

Table 1. displays the summary of all the shear punch tests conducted as a part of this study, including control materials. Red rows correspond to suspect thickness measurements, and blue rows correspond to corrected data using thickness from other samples made from the same material. Often there is the case of multiple corrected measurements for the same sample due to some uncertainty to which sheet of material the sample originated in. The original sheet thicknesses (prior to 3mm x 0.25mm being punched from them) are shown in Appendix A for reference. Further work is needed to correlate individual samples to originating sheet so final corrections can be made. The variation in thickness across a polished sheet is generally small, but sheet to sheet variation is quite significant in some cases.

Table 1: Summary of all 3mm shear punch tests from initial Bor60 irradiation capsule, including some reference controls and ACO3 Duct material.

Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
				C	dpa	mm	MPa	(MPa)	
5/20/16	Cu-1	Annealed OFE Copper	None	-	-	0.255	127	185.3	Control for nanoindation of SP damage
5/20/16	Cu-2	Annealed OFE Copper	None	-	-	0.249	121	184.9	Control for nanoindation of SP damage
5/20/16	Cu-3	Annealed OFE Copper	None	-	-	0.255	125	189.5	
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
4/12/16	HT9 INL-1	HT-9 INL Heat	None	-	-	0.250	380	594.4	Control for nanoindation of SP damage
4/12/16	HT9 INL-2	HT-9 INL Heat	None	-	-	0.251	360	592.2	Control for nanoindation of SP damage
4/12/16	HT9 INL-3	HT-9 INL Heat	None	-	-	0.250	390	600.7	Control for nanoindation of SP damage
12/15/16	C76	HT9 CONTROL 84425	Control	-	-	0.249	410	578.0	Bor60 Control
12/15/16	C59	HT9 CONTROL 84425	Control	-	-	0.245	358	553.0	Bor60 Control
8/25/16	Po33-C23	HT-9	Bor60	~400	~16	0.263	420	578.5	Suspicious Thickness
8/25/16	Po33-C23-C	HT-9	Bor61	~400	~16	0.241	440	631.0	Corrected thickness to other P033 sample
8/25/16	Po33-C23-C2	HT-9	Bor61	~400	~16	0.229	460	664.0	Corrected thickness to other P027 Sample
1/23/17	P033-C61	HT9	Bor60	~400	~16	0.241	480	669.0	Good micrometer
8/25/16	P027-C00	HT-9	Bor60	~375	~16	0.229	510	716.7	
1/23/17	P027-C03	HT9	Bor60	~370	~16	0.230	520	726.0	Good Micrometer
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	
11/4/16	4A1(1)	HT9	ACO3	470	110	0.238	370	521.0	
11/4/16	4A1(2)	HT9	ACO3	470	110	0.220	377	548.0	
11/4/16	5A1(1)	HT9	ACO3	450	155	0.262	315	457.0	
11/4/17	5A1(2)	HT9	ACO3	450	155	0.243	375	542.0	
1/19/17	5A1(3)	HT9	ACO3	450	155	0.239	371	525.0	
1/19/17	5A1(4)	HT9	ACO3	450	155	0.237	377	566.0	
11/4/16	6A1(1)	HT9	ACO3	420	115	0.220	398	555.0	
11/4/16	6A1(2)	HT9	ACO3	420	115	0.241	380	527.0	
11/4/16	6A5(1)	HT9	ACO3	370	15	0.233	467	610.0	
11/4/16	6A5(2)	HT9	ACO3	370	15	0.248	475	608.0	
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	
	ctrl#3A	HT9-ACO3	CONTROL	-	-	0.273	359	576.0	
	ctrl#3b	HT9-ACO3	CONTROL	-	-	0.260	317	572.0	
	HTLD-4A	HT9-ACO3	AC03	505	4	0.263	379	571.0	
	HTLD-4B	HT9-ACO3	AC03	505	4	0.261	346	566.0	
	LTLD-3A	HT9-ACO3	AC03	383	28	0.262	504	667.0	
	LTLD-3B	HT9-ACO3	AC03	383	28	0.248	510	680.0	
	MTHD-1A	HT9-ACO3	AC03	443	155	0.258	351	601.0	
	MTHD-1B	HT9-ACO3	AC03	443	155	0.250	373	598.0	
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
1/24/17	P027-M11	Fe12Cr	Bor60	~370	~16	0.230	360	510.0	
1/24/17	P033-M36	Fe12Cr	Bor60	~400	~16	0.228	360	512.7	
9/15/16	P033-M35	Fe12Cr	Bor60	~400	~16	0.292	320	442.2	Suspicious Thickness
9/15/16	P033-M35-C	Fe12Cr	Bor60	~400	~16	0.228	394	566.0	Corrected thickness to other P033 sample
9/15/16	P027-M10	Fe12Cr	Bor60	~370	~16	0.297	305	418.2	Suspicious Thickness
9/15/16	P027-M10-C	Fe12Cr	Bor60	~370	~16	0.230	385	540.0	Corrected thickness to other P027 sample
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
8/25/16	P033-T27	T91	Bor60	~400	~16	0.229	372	539.6	
8/26/16	P027-T41	T91	Bor60	~370	~16	0.279	370	487.8	Suspicious Thickness
8/26/16	P027-T41-C	T91	Bor60	~370	~16	0.248	420	548.0	Corrected thickness to other P027 sample
8/26/16	P027-T41-C2	T91	Bor60	~370	~16	0.229	440	594.0	Corrected thickness to other P033 Sample
1/23/17	P033-T28	T91	Bor60	~400	~16	0.239	390	556.0	
1/23/17	P027-T02	T91	Bor60	~370	~16	0.248	445	583.0	
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
12/16/16	M84	Fe9Cr	CONTROL	-	-	0.231	213	415.5	
12/16/16	M85	Fe9Cr	CONTROL	-	-	0.232	212	402.9	
1/24/17	P033-M21	Fe9Cr	Bor60	~400	~16	0.218	265	436.0	
9/15/16	P033-M19	Fe9Cr	Bor60	~400	~16	0.219	280	424.4	
1/24/17	P027-M02	Fe9Cr	Bor60	~370	~16	0.243	300	458.7	
9/15/16	P027-M00	Fe9Cr	Bor60	~370	~16	0.287	270	376.0	Suspicious Thickness
9/15/16	P027-M00-C	Fe9Cr	Bor60	~370	~16	0.243	310	444.0	Corrected thickness to other P027 sample
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
1/25/17	P033-N28	NF616	Bor60	~400	~16	0.263	395	584.0	
9/15/16	P033-N27	NF616	Bor60	~400	~16	0.250	410	574.9	
9/13/16	P033-Y25	14YWT - SM13	Bor60	~400	~16	0.251	735	1003.6	via David Hoelzer ORNL
9/13/16	P027-Y04	14YWT - SM13	Bor60	~375	~16	0.271	740	951.4	via David Hoelzer ORNL
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
9/19/16	P027-H02	800H	Bor60	~375	~16	0.271	410	665.3	Suspicious
9/19/16	P027-H02-C	800H	Bor60	~375	~16	0.258	475	698.0	Corrected
1/25/17	P027-H03	800H	Bor60	~375	~16	0.258	470	680.0	
12/16/16	H30	800H-35175	CONTROL	-	-	0.260	150	455.0	Odd elastic loading
12/16/16	H41	800H-35175	CONTROL	-	-	0.260	138	459.0	Odd elastic loading
9/19/16	P027-A02	Fe21Cr32Ni	Bor60	~375	~16	0.259	348	490.7	Suspicious
9/19/16	P027-A02-C	Fe21Cr32Ni	Bor60	~375	~16	0.245	360	519.0	Corrected
1/25/17	P027-A35	Fe21Cr32Ni	Bor60	~375	~16	0.245	370	524.0	
Date	Sample ID	Material	Irradiation	Tirr	Dose	thickness	1% Offset Shear Yield	Shear UTS	Notes
12/15/16	F19	α-Fe	CONTROL	-	-	0.294	225	294.0	
12/15/16	F50	α-Fe	CONTROL	-	-	0.291	232	295.0	
9/19/16	P033-F16	α-Fe	Bor60	~400	~16	0.291	197	290.4	
1/24/17	P033-F17	α-Fe	Bor60	~400	~16	0.295	195	297.0	
9/19/16	P027-F00	α-Fe	Bor60	~370	~16	0.291	215	301.2	
1/24/17	P027-Fzz	α-Fe	Bor60	~370	~16	0.288	215	312.0	Could not fully read sample ID

Data from HT9 shear punch testing from this irradiation, along with samples of similar dose and irradiation conditions tested from the ACO3 duct [3] can be seen in Figure 4. The BOR 60 and ACO3 samples are from the same original lot of HT9 material, 84425. As expected, it appears that irradiation temperature is more significant than dose with respect to their effect on mechanical properties. The ACO3 A plate samples may be outliers due to estimated dose and temperature, vs. more precise calculations for the ACO3 E plate samples. Additional uncertainty of irradiation temperature for the BOR 60 irradiated samples, may also affect the comparison. The control data is quite consistent, as seen in Figure 5.

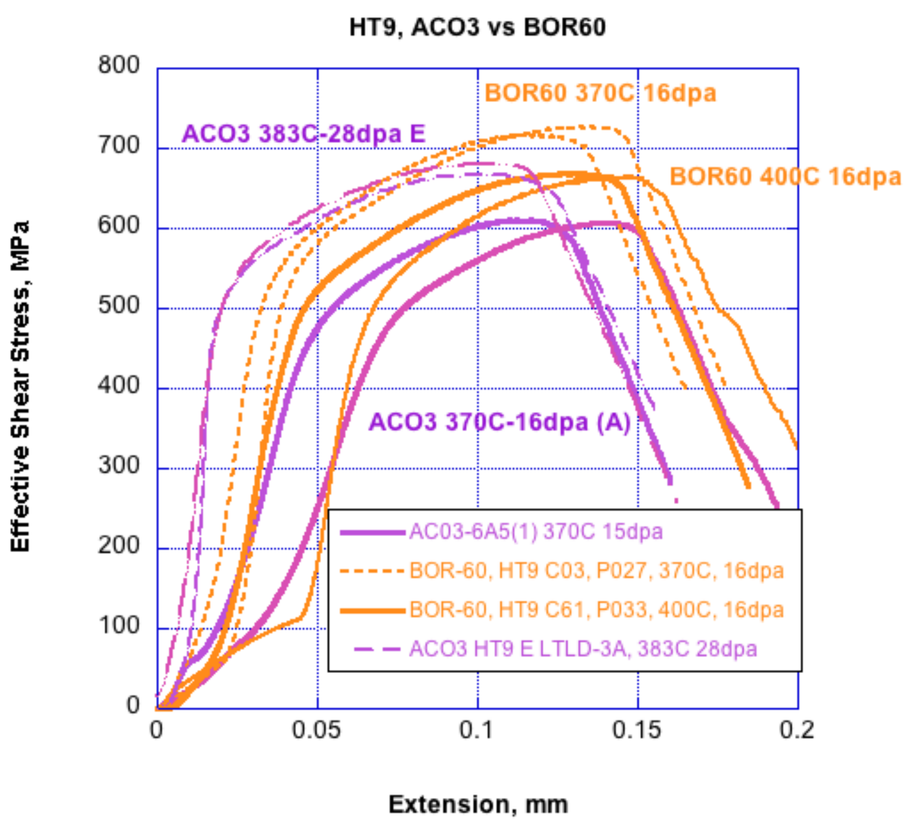

Figure 4. HT9 shear punch data from AC03 and BOR-60 irradiated samples

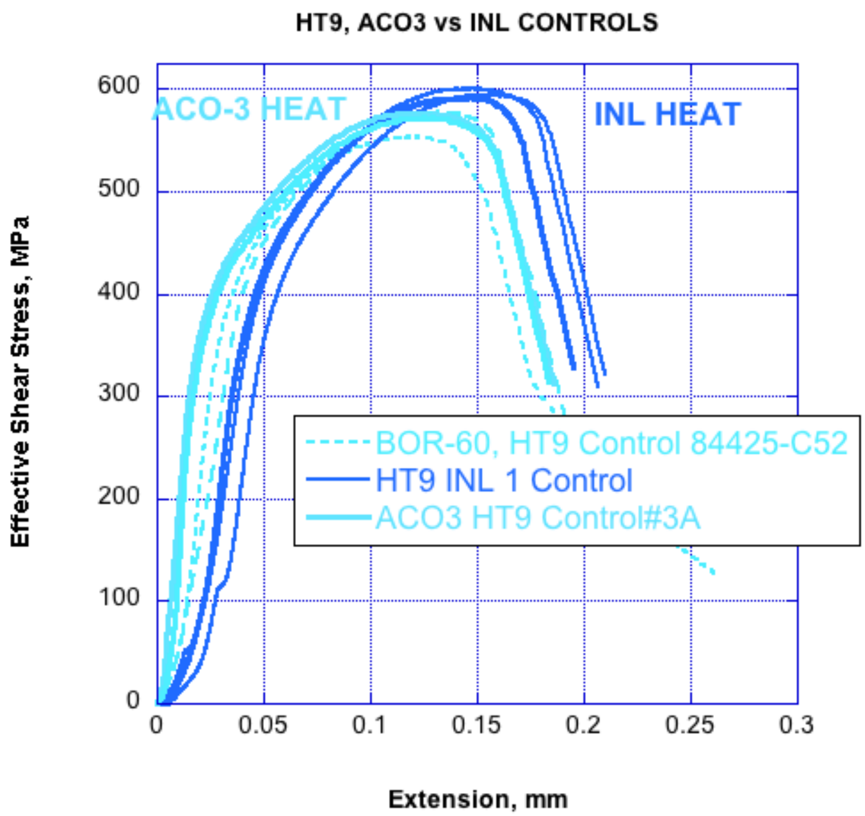

Figure 5. HT9 control shear punch data from AC03, BOR-60 (same as ACO3 lot) and INL heats.

Additional ESS vs. Extension Curves can be seen in Appendix B. Data sets will be more significant with addition of higher dose data from BOR-60 irradiations, along with better thickness corrections and data from other irradiations. Notably, some data sets are missing control samples, which should be measured in the next round of testing.

4. CONCLUSIONS

Despite some uncertainty in the thickness measurements, the results are relatively consistent and are a great basis for comparison for the impending 32 dpa and higher dose TEM samples from the same irradiation (32 dpa sample arriving at LANL later this year). Some additional investigations are ongoing to correlating the individual samples to the original sheet ID's, thus narrowing down the uncertainty of the pretest sample thickness. These samples will be an excellent basis for comparison to the 8mm shear punch samples that are being tested from the ATR NSUF UCSB irradiation. Comparing the controls of each geometry should provide a comparison of size effects for 1mm punchouts in 0.25mm thick samples vs 1.5mm punchouts in 0.5mm thick samples. The irradiated data will fill in some lower dose gaps in the BOR60 TEM shear punch data set. Punches from these samples were sent to UC Berkeley for subsequent nanohardness testing as well as FIB preparation of TEM and APT samples. The complete data set of shearpunch, nanohardness and microstructural data will help inform modelling efforts of irradiated materials properties as a part of a collaboration lead by UC Santa Barbara.

References

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[2] 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

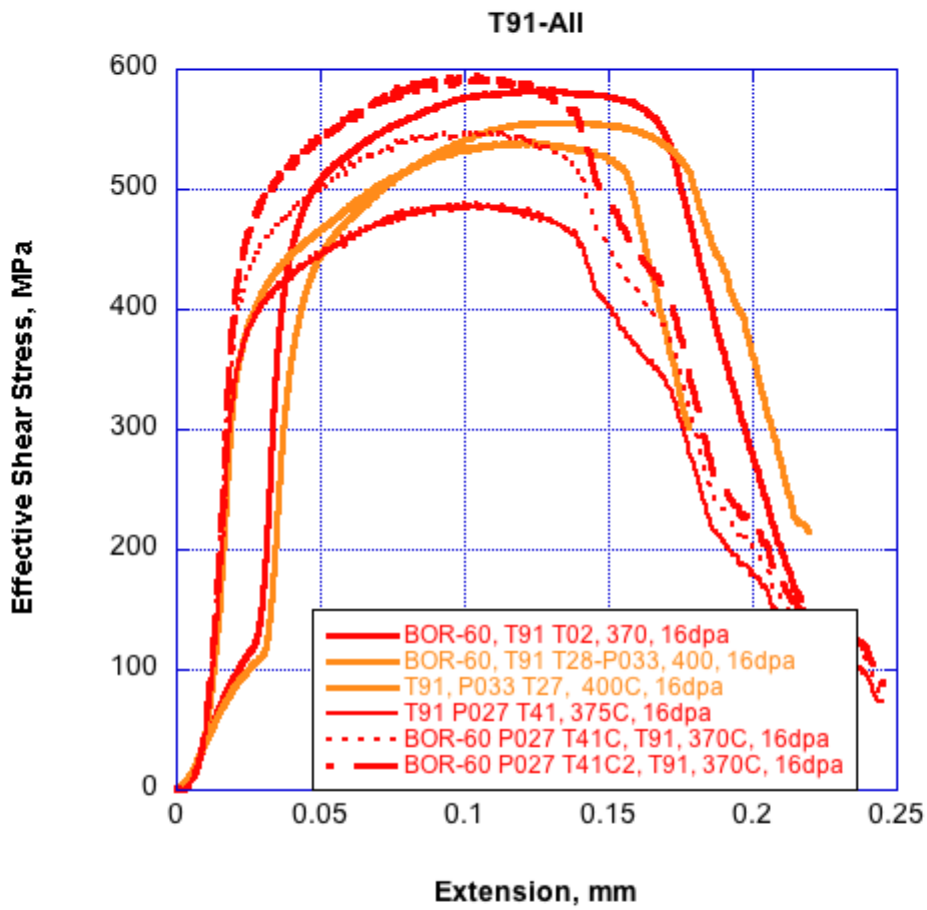
[3] S.A. Maloy, T.J. Romero, P. Hosemann, M.B. Toloczko, Y. Da, Shear punch testing of candidate reactor materials after irradiation in fast reactors and spallation environments, Journal of Nuclear Materials Volume 417, Issues 1–3, 1 October 2011, Pages 1005–1008

Appendix A

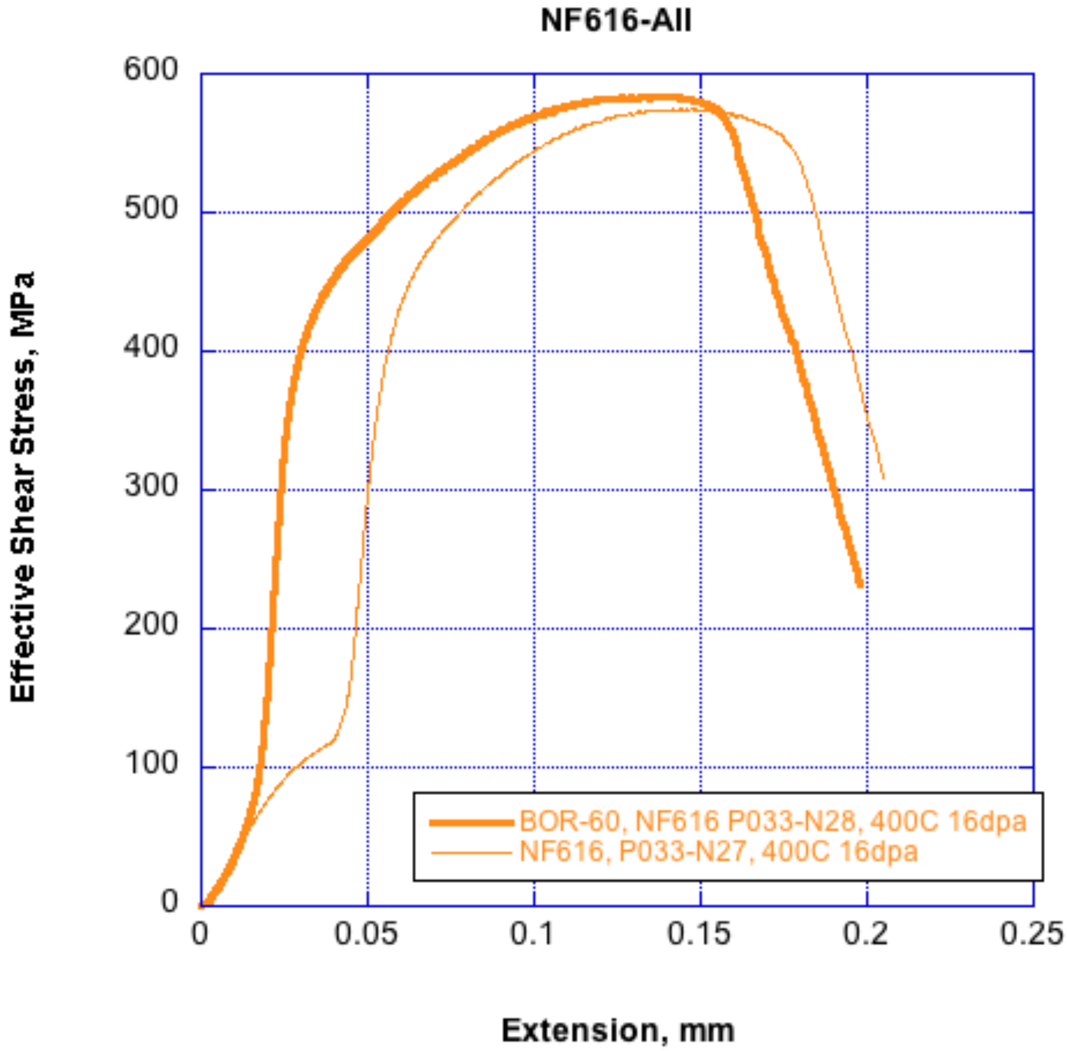
Original Sheet Thickness Measurements

Alloy	Piece	Size (mm)	Thickness 1 (microns)	Thickness 2 (microns)	Thickness 3 (microns)	Average piece	St Dev Piece	All Average	All StDev	note
T91	1	20x25	246	242	238	242.0	4.0	237.22	4.32	
	2	20x25	235	233	235	234.3	1.2			
	3	20x25	234	234	238	235.3	2.3			
NF616	1	34x18	246	253	262	253.7	8.0	252.89	4.62	
	2	34x18	253	253	247	251.0	3.5			
	3	34x18	254	255	253	254.0	1.0			
14YWT-IAEA	1	25x17	250	252	252	251.3	1.2	251.08	3.12	
	2	25x17	254	254	256	254.7	1.2			
	3	25x17	252	252	251	251.7	0.6			
	4	25x17	246	248	246	246.7	1.2			
21Cr32Ni	1	30x10	252	257	257	255.3	2.9	255.78	5.25	
	2	30x10	263	252	260	258.3	5.7			
	3	30x10	252	255	260	255.7	4.0			
	4	30x10	259	242	256	252.3	9.1			
	5	30x10	261	249	256	255.3	6.0			
	6	30x10	263	257	253	257.7	5.0			
800H	1	29x7	254	254	259	255.7	2.9	257.87	3.11	
	2	29x7	262	260	262	261.3	1.2			
	3	29x7	259	260	260	259.7	0.6			
	4	29x7	258	259	259	258.7	0.6			
	5	29x7	252	254	256	254.0	2.0			
9Cr	1	32x20	261	249	268	259.3	9.6	241.22	19.13	
	2	32x20	238	229	235	234.0	4.6			
	3	38x20	244	245	202	230.3	24.5			thin unpolished area on 1/2
12Cr	1	54x20	251	257	246	251.3	5.5	249.50	5.39	
	2	54x20	243	254	246	247.7	5.7			thin unpolished area in middle
ACO3	1	12.5d	230			230.0		227.30	16.75	
	2	12.5d	227			227.0				
	3	12.5d	223			223.0				
	4	12.5d	224			224.0				
	5	12.5d	230			230.0				
	6	12.5d	234			234.0				
	7	12.5d	254			254.0				One side polished
	8	12.5d	244			244.0				One side polished
	9	12.5d	216			216.0				One side polished
	10	12.5d	191			191.0				One side polished
Alpha Fe	1	50x50	290	295	300	295.0	5.0	294.50	3.94	
	2	50x50	298	293	291	294.0	3.6			

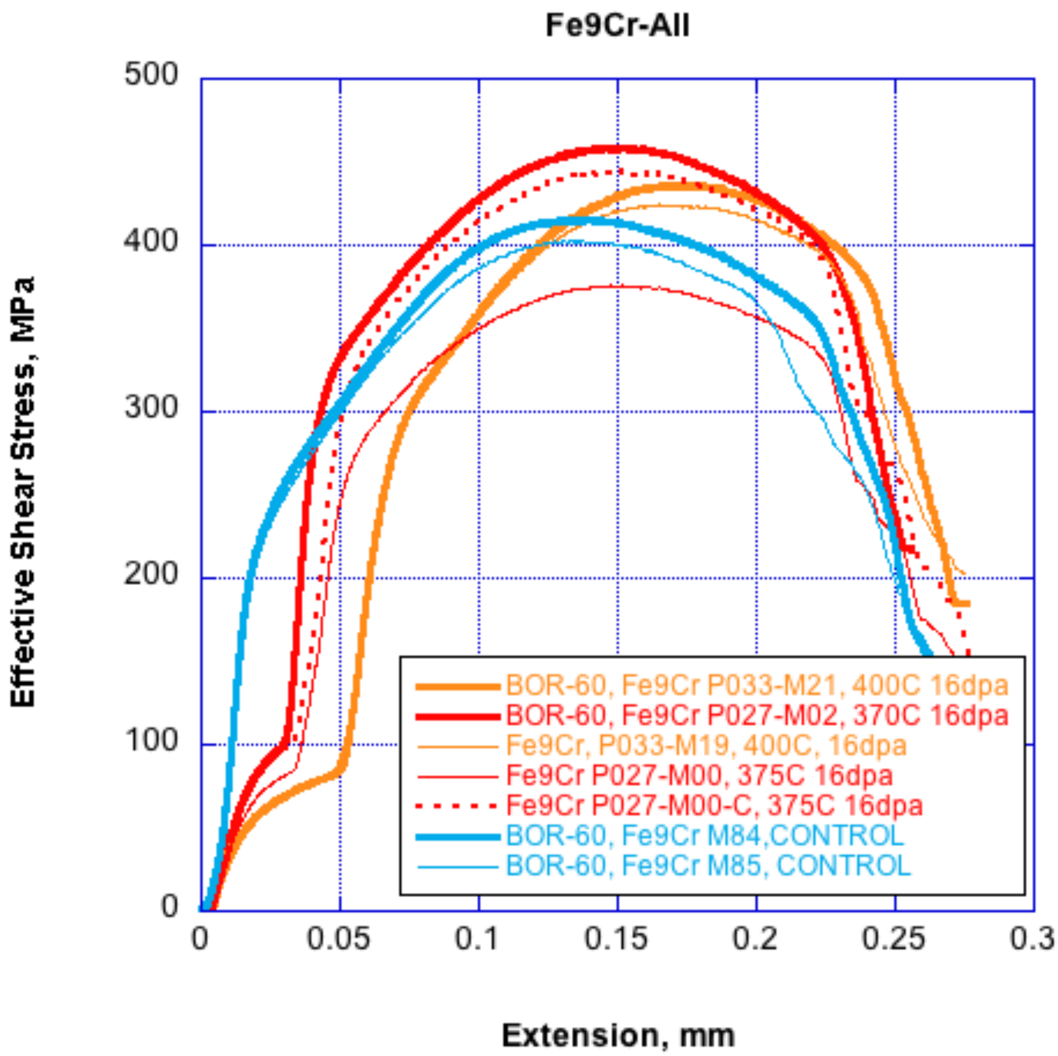
Appendix B
All ESS vs Extension Curves for Initial BOR-60 Irradiated TEM Samples



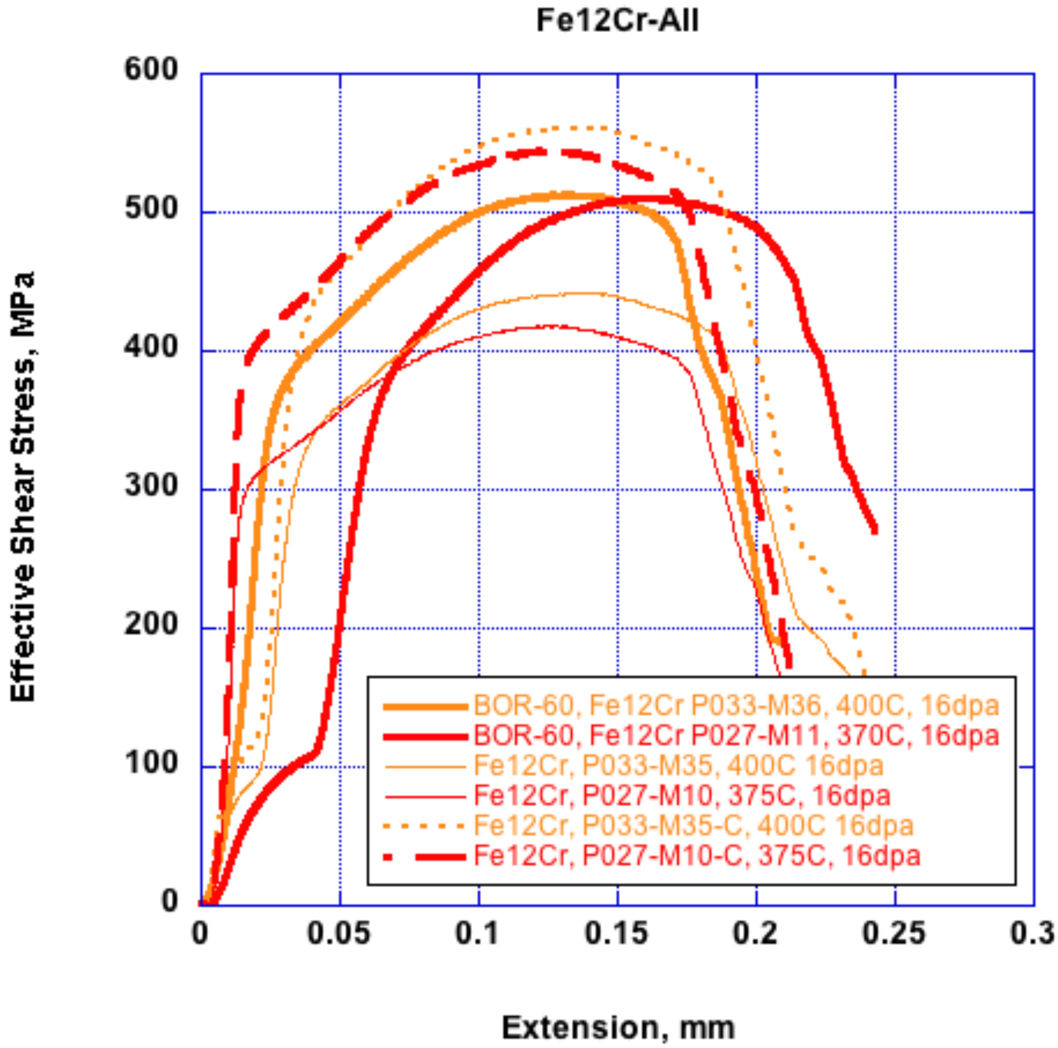
All T91 Data. No control samples available at this time.



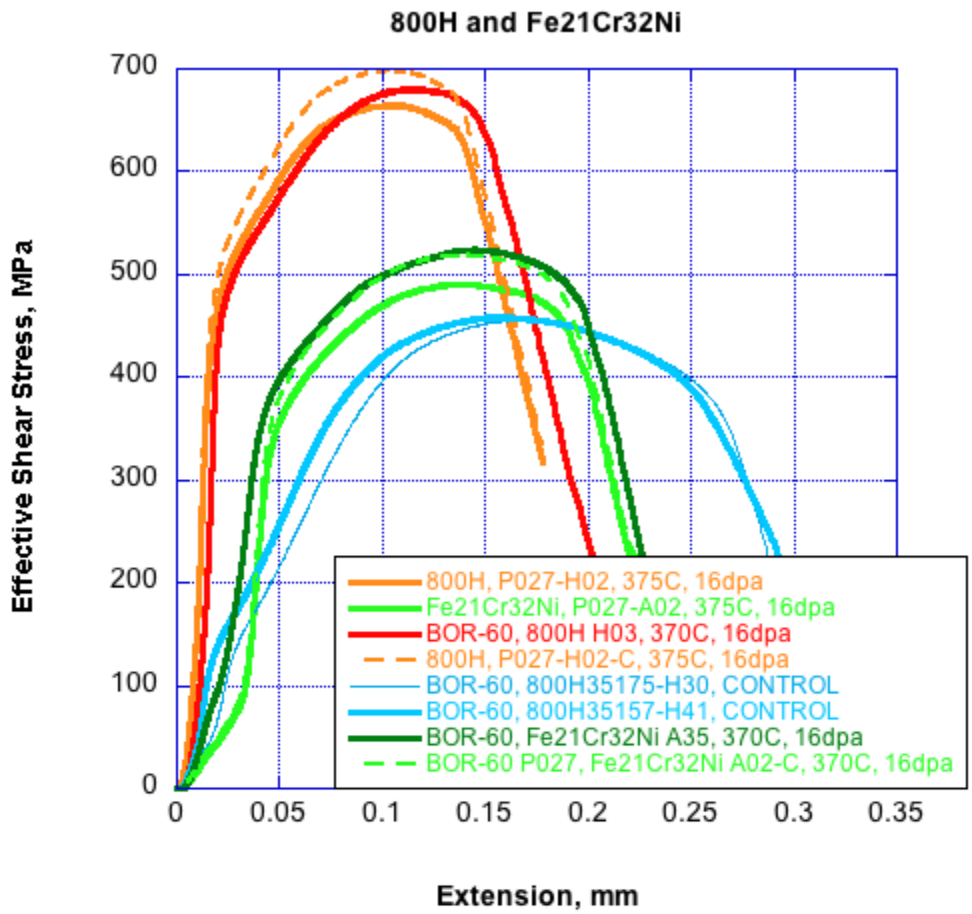
NF616 Samples, no control samples available.



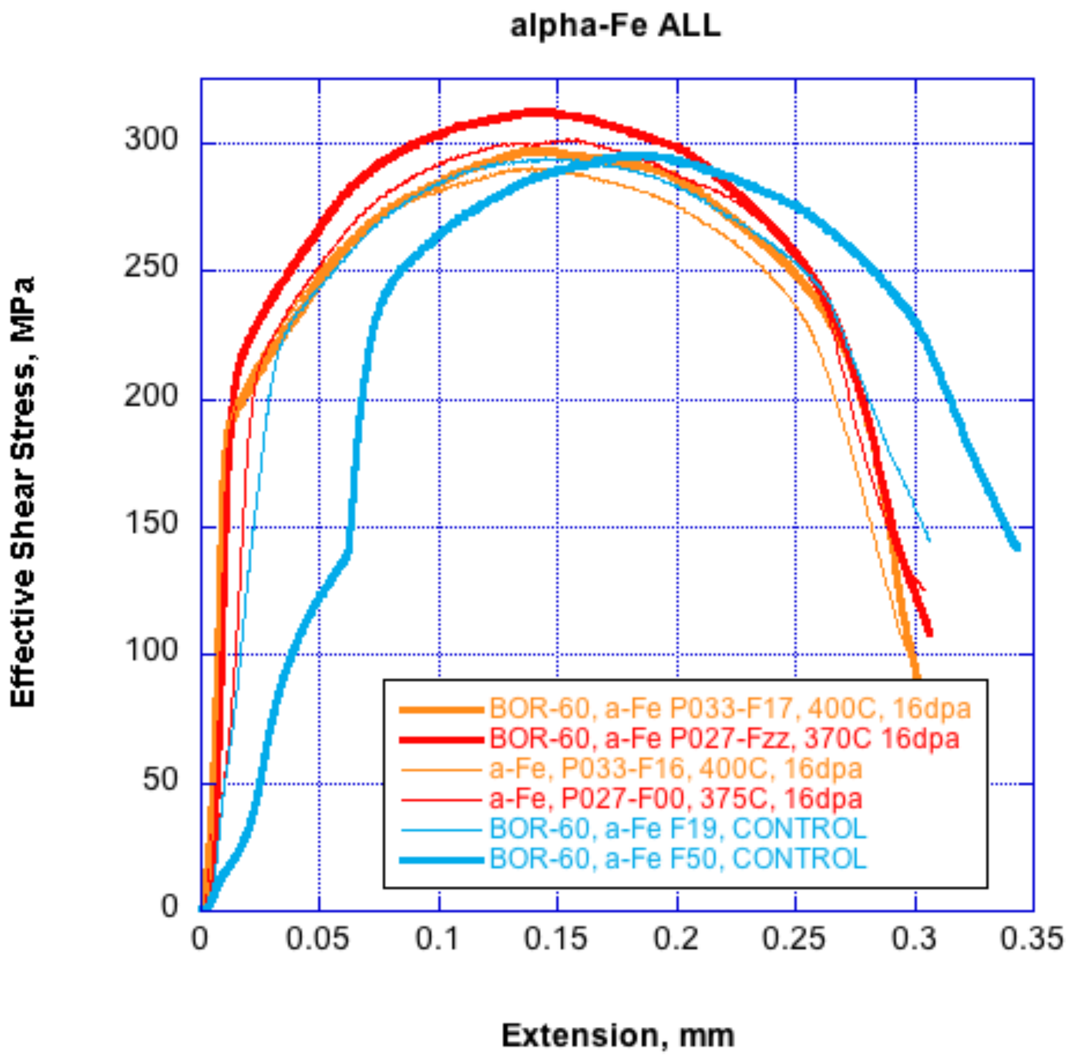
All Fe9Cr Samples



All Fe12Cr Samples, no controls available.



800H and Fe21Cr32Ni samples from BOR60 irradiation, no controls for Fe21Cr32Ni samples



All α Fe samples. Note soft elastic region in F50 control, probably due to cupped sample.