MICROSTRUCTURAL EXAMINATION OF IRRADIATED ISOTOPICALLY TAILORED FERRITIC/MARTENSITIC STEELS FOLLOWING SHEAR PUNCH TESTING

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OBJECTIVE

The objective of this effort is to provide understanding of the effect of hydrogen and helium production during irradiation on post-irradiation mechanical properties in Ferritic/Martensitic steels to be used for first wall applications in a fusion reactor based on microstructural examination.

SUMMARY

Single variable experiments are being conducted to study effects of H/He/dpa on properties based on isotopically tailored alloys. ^{54}Fe has been used to prepare an isotopically tailored duplicate of the commercial steel F82H, and a small number of TEM disks have been irradiated in order to study radiation embrittlement. From single disk specimens, mechanical properties were obtained using a shear punch technique that produces a 1 mm blank from the 3 mm disk. The 1 mm blanks have been thinned and examined by TEM. The novel thinning procedures are described and microstructural observation presented in detail. Little effect of helium on microstructure was found, either in ^{54}Fe isotopically tailored specimens or in a boron doped specimen.

PROGRESS AND STATUS

Introduction

A concern in developing structural materials for fusion power systems is the consequences of transmutation-induced helium and hydrogen on material properties. For the advanced ferritic steel fusion materials option, helium (in appm) will be generated at about ten times the dpa rate, and hydrogen approximately ten times more rapidly than helium. Experiments to define the effect of helium remain controversial[1,2] but severe effects of helium accumulation on fracture toughness have been claimed.[3,4] It is therefore very important to evaluate these transmutation effects in order to establish whether steels can be successfully adapted for fusion applications.

Single variable experiments have been conducted to study effects of H/He/dpa on properties by preparing isotopically tailored alloys. Initially, alloys containing small additions of different nickel isotopes were studied,[5] but the present approach considers alloys made from iron isotopes in order to vary H/He/dpa rates.[6] The controlling reactions are:

^{54}Fe (n,p) ^{54}Mn

^{54}Fe (n,γ) ^{55}Fe; ^{55}Fe (n,α) ^{52}Cr or ^{55}Fe (n,p) ^{55}Mn (n,γ) ^{56}Fe.

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^{54}Fe was used to prepare an isotopically tailored duplicate of the commercial steel F82H,[7] and, because of cost and irradiation space limitations, a small number of TEM disks were irradiated in order to study radiation embrittlement.

Very recently, individual TEM disks were made available for post-irradiation examination. It was possible to obtain mechanical properties from three alloy conditions for which only a single disk was available, and from a fourth condition where several disks were available in order to compare with results on unirradiated controls. Mechanical properties were obtained using a shear punch technique that produces a 1 mm blank from the 3 mm disk, and microstructural information was obtained from the 1 mm blanks thinned to electron transparency. The purpose of this paper is to document the experimental technique used in preparing TEM specimens and to describe microstructural observation.

Experimental Procedure

A 4 g batch of isotopically tailored F82H ferritic/martensitic steel was prepared as described previously,[7] and specimens 3 mm in diameter, intended for transmission electron microscopy (TEM), were obtained along with a specimen of standard F82H and a specimen of boron-doped F82H, following irradiation in either the JP17 irradiation experiment to 2.3 dpa at 250°C or the JP22 irradiation experiment to 34 dpa at 300°C in the High Flux Isotope Reactor (HFIR) in Oak Ridge, TN.[8-9] Also available were unirradiated control specimens of the isotopically tailored alloy, the standard alloy and another heat of the steel, designated the IEA heat.[10] F82H has the approximate composition (in weight %) Fe-7Cr-2W-0.2V-0.1C-.04Ta-0.01N. Composition details are provided in Table 1 and irradiation details are given in Table 2.

Table 1. Composition of alloys of interest.

Alloy	Cr	C	W	V	Ta	B	Mn	N	Si
F82H ^{54}Fe	7.1	0.097	1.8	0.17	0.04	-	0.4	0.007	0.55
F82H STD	7.46	0.097	2.1	0.18	0.03	0.0004	0.07	0.004	0.09
F82H ^{10}B	7.25	0.098	2.1	0.22	0.04	0.0058	0.5	0.002	0.17

Room temperature mechanical properties information was obtained using a shear punch technique that produces 1 mm blanks from 3 mm disks.[12] Fourteen shear punch tests were performed comprising nine control tests and five tests on irradiated samples. The results of shear punch testing are reported elsewhere.[12]

Preparation of miniature TEM specimens has been a goal for many years because of the inherent benefits arising from reduced magnetic moment and reduced radioactivity. However, previous attempts were not completely successful, either because the smaller specimens were punched from thinned TEM samples, causing excessive deformation, or specimens were prepared from disks smaller than 3 mm, requiring nickel or chrome plating before thinning in order to increase the sample size for simplified handling. Also, it was found that the composite samples tended to come apart during examination because bonding was not ideal. The present effort provided an alternative approach, based on the observation that 1 mm shear punch blanks were free of surface buckling and with well defined cylindrical shear surfaces. Finite element analysis supported the view that 1 mm blanks would provide acceptable material

Table 2. Irradiation conditions for TEM samples.

ID	MATERIAL	CONDITION*
A943-5	F82H IEA	Unirradiated
F191-3	F82H STD	Unirradiated
C103	F82H STD	300°C, 34 dpa, 21.8 appm He
FN91-3	F82H ^{54}Fe	Unirradiated
FN51-2	F82H ^{54}Fe	250°C, 2.3 dpa, 6 appm H, 4.5 appm He
C603	F82H ^{54}Fe	300°C, 34 dpa, 650 appm H, 65.3 appm He
C203	F82H ^{10}B	300°C, 34 dpa, 321 appm He

* H values are predicted, but He values are measured.[11]

for microstructural examination, because the stress state induced by punching at the center of the 1 mm blank remained well below yield.[13] Also, the punching process could be used to provide a 3 mm support disk and then thinning could be accomplished by standard procedures. The need for a glue joint was apparent, but superglue did not allow sufficient positioning control to work properly.

Therefore, specimens for TEM were prepared from the punched 1 mm blanks by pressing them into 3 mm punched 316 stainless steel disks and using thermal setting epoxy to ensure good bonding. Surfaces were then ground to remove excess epoxy and samples were electropolished to perforation in a standard Tenupol twin jet polishing apparatus. The epoxy bond was found to be resistant to chemical attack and provided a good bond as well as a supporting rim for the thin foil adjacent to the perforation. Examples of an unpunched disk, a punched disk with 1 mm blank and a thinned 1 mm specimen in a 3 mm stainless steel disk are shown in Figure 1.

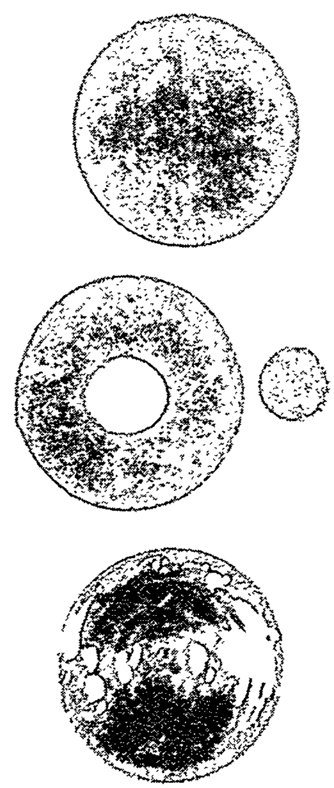

Figure 1. Sample Configurations for TEM Specimen Preparation.

Results

TEM specimen preparation was successful for all seven conditions available. However, the control condition for the IEA heat of F82H was not examined in this study. All thin foils prepared from the 1 mm blanks could be considered very good to excellent, with negligible bending and sufficient thin area at the center of the disk. No problems were encountered due to magnetic interactions or excessive radioactivity. Examples of the microstructures at low magnification are shown in Figure 2. Micrographs have been selected to show prior-

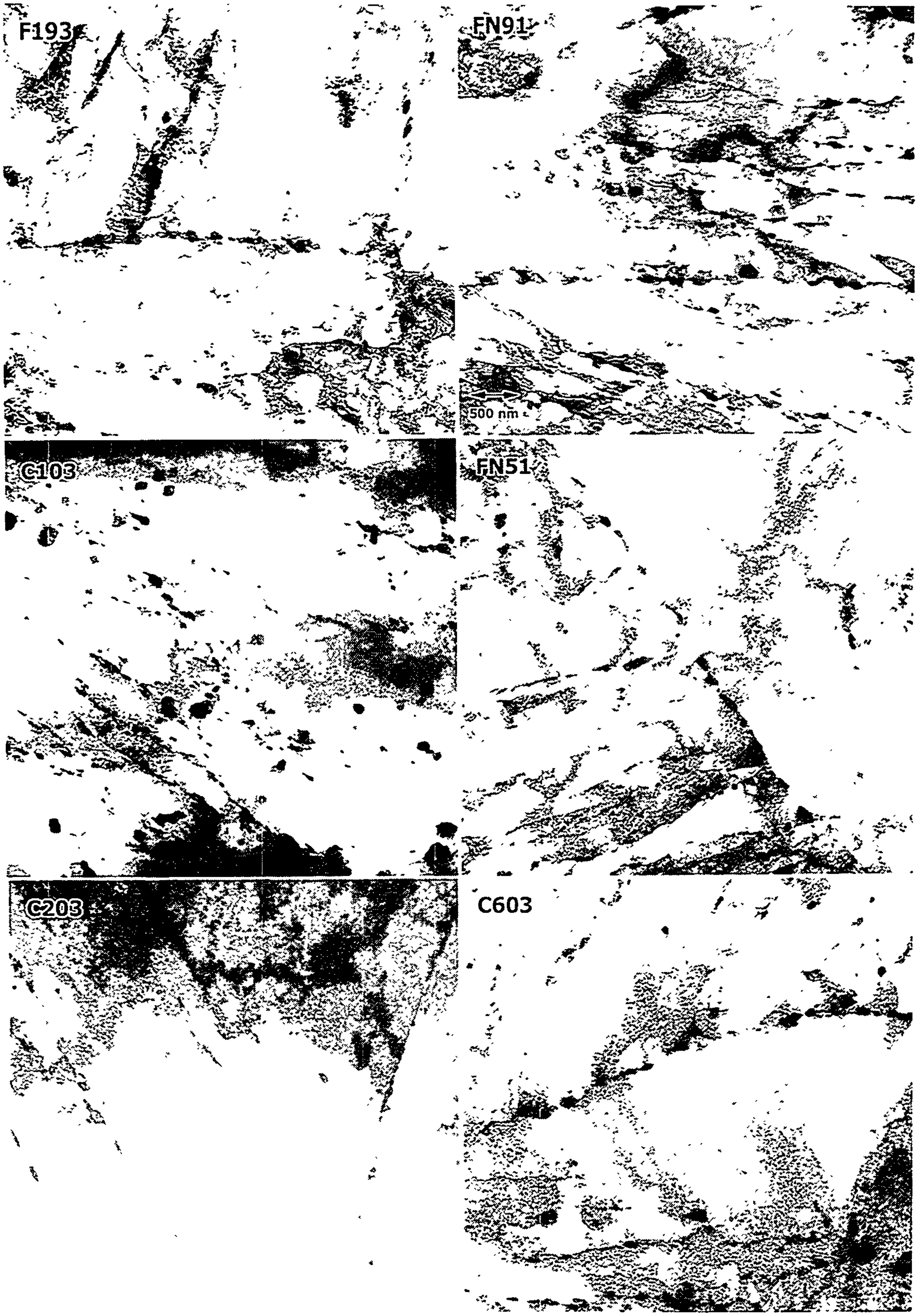

Figure 2. Microstructures at low magnification.

austenite boundary structure when they could be found, and are arranged with standard F82H on the upper left, ^{54}Fe F82H on the right with increasing dose from top to bottom, and ^{10}B F82H at the bottom left. Figure 2 demonstrates that foils provided abundant thin area. However, foils of specimens C103 and C203 had limited thin area compared to the other conditions. From Figure 2, it can be shown that all conditions appear typical of F82H. Lath boundaries are decorated with carbides and dislocation structures tend to be subgrain boundaries in the unirradiated conditions: F193 and FN91. Figure 2c of specimen C103 shows coarser carbide structure than the control condition of F193 in Figure 2a, but differences should probably be ascribed to area-to-area differences in the carbide distribution. The effect of irradiation is seen as damage on a fine scale that tends to obscure subgrain structure.

In order to examine the fine scale damage due to irradiation, the microstructures were imaged under conditions approaching weak beam using $\vec{g}$ = [200] and [01$\bar{1}$] for foils oriented near (011). Dislocation microstructures for the six conditions are given in Figure 3. Figure 3 is arranged to show [01$\bar{1}$] dark field contrast images with $\vec{g}$ vertical for all conditions, and in the three cases where two images dark field images for a given condition are provided, a 200 dark filed image is provided on the left (3c, 3e and 3g) with $\vec{g}$ horizontal. Therefore, Figure 3 provides examples of the unirradiated microstructures for F82H STD in a and F82H ^{54}Fe in b, F82H ^{54}Fe is then shown with increasing dose in c, d e and f, and then F82H STD in g and h can be compared with F82H ^{10}B in i and j for the high dose condition. Figure 3i presents a bright field image in order to assess cavitation development in F82H ^{10}B. From Figure 3, it can be noted that dislocation structure prior to irradiation consists of subgrain boundaries and a low density of straight dislocations within laths. Following irradiation, fine dislocation structure develops consisting of both $\frac{a}{2}$<110> and a<100>. [Under [200] contrast, strong vertical images show one set of a<001> and all other images are of $\frac{a}{2}$<111> type with all sets visible.] In F82H ^{54}Fe, irradiation to 2.3 dpa at 250°C produces $\frac{a}{2}$<111> dislocations with loops as large as 20 nm, as well as a few a<100> loops as large as 13 nm. Irradiation to 34 dpa increases both the total dislocation density and the fraction of a<100> dislocations. In comparison, the F82H STD and F82H ^{10}B at 34 dpa produce microstructures similar to F82H ^{54}Fe. The bright field image of F82H ^{10}B irradiated to 34 dpa reveals no significant cavitation despite the He level at 321 appm He. Therefore, irradiation of this isotopically tailored series of alloys at 250 to 300°C develops both $\frac{a}{2}$<110> and a<100> dislocation structure and this structure should be responsible for changes in mechanical properties due to irradiation.

Examination included limited efforts at FEG-STEM compositional analysis. Compositional analysis of carbide precipitates was possible and sufficiently detailed to demonstrate transmutation of approximately a third of the W to Os, with no evidence for the intermediate product, Re, following irradiation of standard F82H to 34 dpa. This can be shown from Figure 4, which provides x-ray spectra for a carbide precipitate in F82H STD irradiated at 300°C to 34 dpa after subtraction of a hole count. The spectrum has been labelled to show peak positions for W, Re and Os. The Os peak is approximately one third of that for W with Re absent. Therefore, it can be concluded that ⅓ of the W has transmuted to Os with little of the intermediate product Re retained, and the Os is retained in the chrome carbides. This transmutation response has been predicted by Garner and Greenwood.[14]

Discussion

This effort demonstrates that it is possible to obtain mechanical property and microstructural information on very limited quantities of material: TEM samples weigh about 0.01 g and the 1 mm blank about 0.001 g. The techniques described above not only allow for experiments

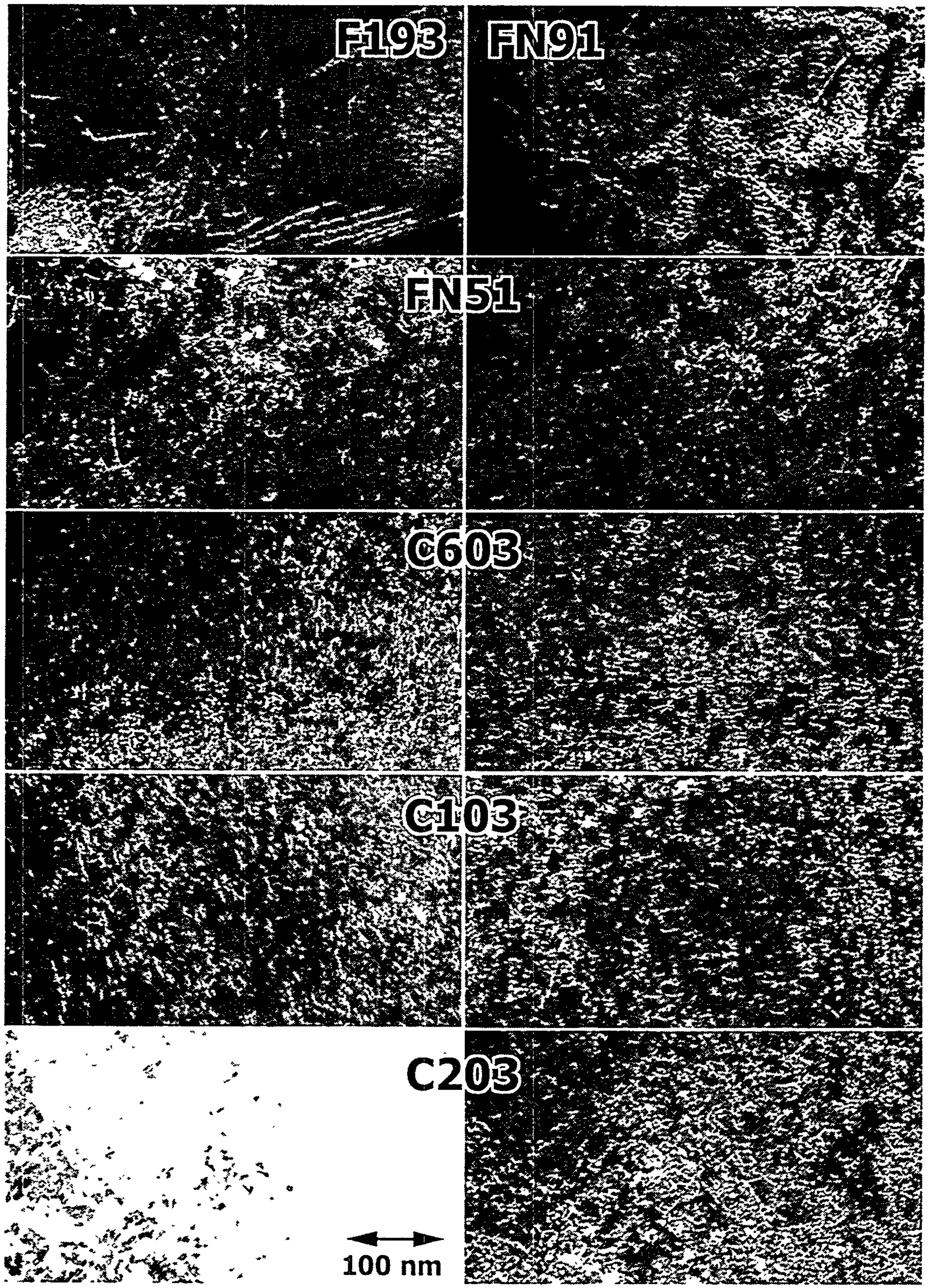

Figure 3. Microstructures at higher magnification showing dislocation structures in dark field a-h and j and in bright field i. See text for details.

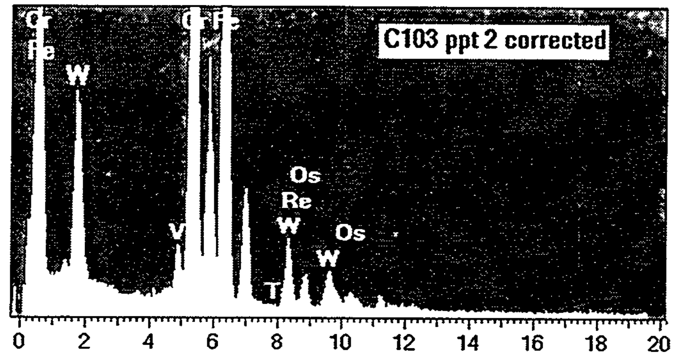

Figure 4. X-ray spectrum for carbide particle in specimen C103 (with background removed).

where material is very limited, very expensive or very difficult to obtain in the desired treatment or irradiation condition, but they also allow sampling of complex conditions. For example, in irradiation creep experiments where strong gradients exist in temperature or flux, it may now be possible to provide understanding of property and microstructural variation over distances of one or two mm. The added advantages of effectively smaller TEM sample volumes that reduce radioactivity or magnetic interactions are also of benefit.

The intent of isotopic tailoring experiments is to assess the effect of hydrogen and helium production during irradiation on properties and microstructure. Because mechanical properties for the different alloys following irradiation at 300°C are very similar, the present results do not demonstrate a significant effect of helium on mechanical properties, and any effect of hydrogen is probably small, although hydrogen production levels are not completely understood. However, it should be noted that concerns about a large effect of helium on ductile brittle transition behavior are centered on irradiation temperatures in the 400°C range. Therefore, the present results are probably not directly pertinent to that issue. However, the absence of precipitate formed during irradiation as found in the these ^{54}Fe isotopically tailored alloys differs from the case where Ni isotopic tailoring was used.[ref]

CONCLUSIONS

Isotopic tailoring is being used to study effects of transmutation on mechanical properties.

Shear punch tests can provide mechanical properties information with limited numbers of TEM samples creating 1 mm blanks that can be successfully prepared for microstructural examination.

Microstructural comparisons show that hardening is due to development of $\frac{a}{2}$<110> and a<100> dislocation structure with little difference between the different alloys following irradiation to 34 dpa at 300°C.

FUTURE WORK

This work will be continued within the confines of funding and specimen availability.

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