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Materials Development

TME - File

EVALUATION OF THREE
CANDIDATE CLUSTER
PLATE MATERIALS

(Title Unclassified)

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EVALUATION OF THREE CANDIDATE CLUSTER PLATE MATERIALS

(Silver-Indium-Cadmium,

Silver-Indium-Cadmium-Tin,

and Boron 10 Containing Stainless Steel)

A. Filippi

October 18, 1963

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EVALUATION OF THREE CANDIDATE CLUSTER PLATE MATERIALS

(Silver-Indium-Cadmium, Silver-Indium-Cadmium-Tin and Boron 10
Containing Stainless Steel)

ABSTRACT

The mechanical properties of three nuclear control materials were evaluated for cluster plate applications. The alloys studied were: 80Ag-15In-5Cd, 75.6Ag-11.5In-8.6Cd-4.4Sn and 1% boron stainless. Annealed 80Ag-15In-5Cd plate displayed an unusually high yield strength which approached that of the tin-containing alloy. The increased yield strength is thought to be a result of the type of fabrication employed and a relatively fine final grain size. The strength of 80Ag-15In-5Cd can be greatly increased through cold work (approximately 50% reduction in thickness), and recrystallization will not occur in 50 hours at 760 °R. Material in this condition does, however, rapidly recrystallize at slightly higher temperatures. The silver-base alloys can hardly be considered for any structural engineering use at temperatures above 800 °R because of appreciable loss of strength.

The strength, ductility and notch properties of 1% boron-containing stainless steel remain relatively high at test temperatures between 140 °R and 1500 °R. Because of this, the alloy is considered the only one of the three candidate materials definitely adequate for cluster plate application.

The possibility of irradiation-induced property change in these alloys was also evaluated. None of the three candidate materials will be seriously affected by the NERVA radiation environment.

INTRODUCTION

A thermal neutron absorber is required at the dome end of the NERVA core to prevent flux peaking from occurring in the upper portion of the core. This need resulted in a decision to use an absorbing material for the cluster plates which compressively load the cluster assemblies.

Three materials, 1.0% gadolinium-containing stainless steel, 1% boron 10-containing stainless steel, and 80% silver-15% indium-5% cadmium have been suggested for cluster plate use. The need for process development work on gadolinium-stainless, and the uncertainty of availability and low temperature mechanical properties of the boron 10 stainless, however, narrowed the choice of materials to the silver-base alloy for use in early reactor tests.

In the meantime, an effort has been made to establish the mechanical properties of both the 80Ag-15In-5Cd and the 1% boron 10 stainless alloys. However, since the available literature indicated that the 80Ag-15In-5Cd alloy is relatively low in strength the study was expanded to include some work on the strengthening effect of cold work and further alloy element additions. Also, since the metallurgical properties of boron stainless depend only on the total boron concentration and not on the isotope concentration, 1% natural boron stainless rather than 1% B¹⁰ stainless was evaluated.

This report does not describe the development currently in progress on other candidate cluster plate materials such as gadolinium-stainless or gadolinium oxide-containing stainless steel. This work will be covered in a future report.

MATERIALS AND PROCESSING

Two silver-base materials were evaluated, one the aforementioned 80 silver-15 indium-5 cadmium, the other a silver-indium-cadmium alloy containing tin as a strengthener. Both alloys were melted and fabricated into 0.150" x 3.500" x 20.0" plates. The fabricating procedures used to produce the plates are outlined in Table I. Wrought plate was ordered to approximate the fabricating conditions which would be used to produce material for cluster plate applications. The specified chemistry asked for on the purchase order, and the actual chemistry on the delivered plates are shown in Table II.

The 1% boron stainless alloy was fabricated by hot rolling into five-inch wide by 50-mil thick strip and was supplied in the fully annealed condition. Chemical composition of the alloy is shown in Table II. The base composition of the alloy conforms roughly to an AISI 305 stainless steel.

EVALUATION OF THE SILVER-BASE ALLOYS

The decision to use 80Ag-15In-5Cd for cluster plates in initial reactor tests was based upon data generated on control material development for pressurized water reactors. Typical data, however represents extrusions and does not cover the temperature range of interest for NERVA application. Some of this data is given in Table III⁽¹⁾. Although this alloy was judged adequate for initial reactor tests, its low yield strength prompted a study of cold worked as well as annealed plate. The cold worked material was reduced approximately 50% in thickness during the final working step and will hereafter be referred to as 50% C.W. Ag-In-Cd (See Table I). The material annealed at 752 °F for one hour will be designated as Annealed Ag-In-Cd. The Ag-In-Cd-Sn alloy was studied in the annealed condition.

Additions of tin to Ag-In-Cd alloys increase the strength of these materials. The increased strength is due in part to the formation of a second hexagonal close packed phase in the structure of the quaternary alloy when the electron-atom ratio (e/a) exceeds 1.4⁽¹⁾. The addition of tin also contributes to increased strength in the single phase face centered cubic alloys when the e/a is 1.4 or lower. The mechanical properties, electron-atom ratio, and composition of several Ag-In-Cd-Sn alloys are shown in Table IV⁽¹⁾. The data show an increasing strength and decreasing ductility with increased e/a. A good compromise of ductility and strength, however, appears to be given by an alloy of e/a between 1.417 and 1.435 (alloys 4 and 5, Y.S.: 33,000 psi, % El: 31%). The nominal composition of the Ag-In-Cd-Sn alloy used in this investigation was chosen such that its electron-atom ratio would lie between the limits set by alloys 4 and 5. Its composition was also chosen so that a variation of ± 0.25 wt.% per element would not result in an e/a which was out of the bounds given by these alloys. The as-received material (Table II) had an e/a of 1.419.

The tensile properties of the annealed Ag-In-Cd, and the Ag-In-Cd-Sn alloy were gathered over the temperature range 140 °R to 1460 °R. This range adequately covers the limits which might be expected under NERVA conditions. For example, the

steady-state operating temperature of the cluster plates is anticipated to be around 300 °R, and decay shut-down temperature will probably not exceed 1000 °R. Tensile properties on the 50% C.W. Ag-In-Cd were gathered from 140 to 750 °R only, since recrystallization was found to occur in short times at temperatures in the vicinity of 800 °R. The tensile specimen used to evaluate the silver-base alloys is shown in Figure 1.

Mechanical Properties of Annealed Ag-In-Cd

Initial mechanical property studies were made both parallel and transverse to the original rolling direction to determine whether any anisotropy existed. All results were similar regardless of direction.

The tensile strength, 0.2% offset yield strength, percent elongation in one inch and percent reduction of area are plotted against test temperature in Figure 2. Each point represents an average of two or more tests, with the exception of the tests at 140 and 1460 °R where only one specimen was evaluated. The mechanical properties of the alloy are relatively constant from 140 to 750 °R. In this range of temperature a tensile strength of about 45,000 psi, and a yield strength of about 20,000 psi with more than adequate ductility is displayed. The tensile and yield strength drop rapidly above 800 °R and the alloy can hardly be considered of any structural value above this temperature.

The yield strength data shown in Figure 2 are abnormally high as compared to that indicated for control rod extrusions (Table III). The increased yield strength might be ascribed to incomplete annealing, but this is doubtful since the tensile strength, hardness (DPH 67) and microstructure (Figure 3, photomicrographs 1 and 2) correspond to fully annealed material. The anomalous yield strength is probably due to more subtle variations in fabrication and grain size. Fabrication of the material used in this investigation involved cold rolling and annealing (Table I) whereas the extrusions were produced in one hot working step. The grain size of the plate was also relatively fine, ASTM 7-8, (Photomicrographs 1 and 2 of Figure 3), and a trend toward an increased yield strength resulting from a small grain size is apparent in the data of Table III.

Mechanical Properties of 50% C.W. Ag-In-Cd

A hardness study as a function of annealing temperature was performed prior to determining the mechanical properties of this material. The study was undertaken to roughly fix the short time (1/2 hour) "recrystallization temperature" of the alloy. Figure 4 shows the results. The decrease in hardness and onset of recrystallization is seen to occur at about 800 °R. Because of this, the tensile evaluation of the cold worked alloy was limited to a maximum temperature of 750 °R.

Initial tensile tests were performed on both longitudinal and transverse samples. Results were similar regardless of direction. A plot of mechanical properties against test temperature is shown in Figure 5. Each point is the average of two results with the exception of the test at 140 °R where only one specimen was evaluated. The data show that a major increase in strength is obtained through cold work with the retention of an adequate amount of ductility. The highly cold worked structure of the alloy is shown in photomicrograph 3, Figure 3.

In order to better establish the elevated temperature limitation of this material an annealing study was performed at various recrystallization temperatures in the range 760 to 860 °R. The results of the study are shown in Figure 6. The data indicate that material in this cold worked condition could be used up to 50 hours at 760 °R without the occurrence of any recrystallization. At slightly higher temperatures, e.g., 835 °R, however, recrystallization becomes rather rapid.

Mechanical Properties of Ag-In-Cd-Sn

The mechanical properties of this material are plotted against temperature in Figure 7. The 140 and 1460 °R results represent only one test. The other results are averages of two tests per temperature. The room temperature ductility met the aim value, but the yield strength did not quite reach the expected 33,000 psi. The ductility of the alloy is also somewhat limited at low temperatures, and at temperatures in the vicinity of 800 °R. In general,

the properties of this material cannot be considered any better than those displayed in Figure 2 for the 80Ag-15In-5Cd alloy.

The microstructure of the alloy is shown in photomicrograph 4 of Figure 3. The two-phase structure is readily apparent in the photomicrograph. Although no x-ray diffraction identification work was performed, it is presumed that the stained, slightly twinned, lower hardness areas, represent the face centered cubic phase, while the light etching, harder areas, show the close-packed hexagonal phase. (The dark rhombic markings are hardness impressions.)

EVALUATION OF 1% BORON STAINLESS

Both smooth and notch bar tensile properties of this alloy were studied over the temperature range 140 to 1500 °R. Notched specimens were included since it was felt that the complex boron-metal second phase, present in boron stainless alloys, might induce notch sensitivity. Both a large-radius notched specimen, $r = 0.020"$ ($K_t 3.1$), and a sharp-notch radius specimen, $r = 0.003"$ ($K_t 7.4$) were used. The photomicrographs in Figure 11 display the structure of the alloy. The complex boron-metal (Fe, Cr and Ni) phase is dispersed as particles in an austenitic matrix. Heavy etching is required to bring out some structure in the matrix, i.e., photomicrograph 3.

Although the material was supplied in the fully annealed condition, specimens were taken parallel and transverse to the rolling direction to determine the degree of anisotropy, if any, introduced by the slight elongation of the second phase during rolling. The configuration of the tensile specimens are shown in Figure 8. All smooth-bar data was gathered on two or more specimens per test temperature. Only one notched specimen was evaluated per test temperature. The notched specimen dimensions were carefully checked on a tool maker's microscope to insure their acceptability.

The smooth bar tensile properties are plotted in Figure 9. The strength and ductility of the alloy remain relatively high regardless of temperature, and no major dependence of properties on test direction is apparent.

The notched properties are plotted in Figure 10. The data indicate that these properties are somewhat anisotropic, since in general the transverse notched strength ratio is lower than the longitudinal notched strength ratio. This is probably a result of the larger cross sectional area of brittle boron-metal second phase being perpendicular to the axis of a transverse specimen as opposed to that area perpendicular to the axis of a longitudinal specimen. This area difference results from slight elongation of the second phase in the rolling (longitudinal) direction during fabrication, and is apparent in the photomicrographs 1 and 2 of Figure 11. (The longitudinal picture represents a view of a plane perpendicular to the axis of a transverse specimen, and the transverse view one perpendicular to the

axis of a longitudinal sample.) In general, however, the degree of anisotropy is slight and can be judged negligible.

The material definitely displays a certain degree of notch sensitivity as judged by a notched strength ratio of less than 1. This ratio is in the range 0.9 to 1 for the large radius (K_t 3.1) sample, and tends toward a value of 1 at low temperatures. This unusual tendency toward an improved notched strength ratio at low temperatures is also displayed by the sharply notched samples (K_t 7.4). Although the alloy is somewhat notch sensitive, its use for cluster plate applications will not involve stresses of high enough magnitude to result in a stress concentration problem. This would be especially true at low temperatures where its strength is much more than adequate for the application. Of the alloys tested the 1% boron stainless displays by far the best combination of mechanical properties over the temperature range 140 to 1500 °R.

IRRADIATION EFFECTS

Neutron irradiation damage in B^{10} stainless may occur by fast neutron-induced displacements and thermal neutron $B^{10} (n, \alpha)$ reaction. The fast neutron flux in the vicinity of the cluster plates is in the order of 7.2×10^{17} nvt for a 20-minute test. This exposure is a good deal lower than the 10^{20} to 10^{21} nvt which results in considerable loss of ductility in austenitic stainless steels⁽²⁾. The thermal flux for such a test is in the order of 2.4×10^{17} nvt. An estimate of the B^{10} burnup from this thermal neutron exposure is much less than 1% of the total boron content of a 1 w/o B^{10} -containing stainless ($< 1\%$ burnup)⁽³⁾. The effect of thermal neutron irradiation on the mechanical properties of 0.93 w/o B^{10} -containing stainless steel is shown in Figure 12⁽⁴⁾. The data show that the estimated NERVA burnup will certainly not impair the mechanical properties of the alloy. Therefore, the alloy is definitely suitable for NERVA hot test cluster plate applications.

The susceptibility of the silver-base alloys to fast neutron induced damage cannot be evaluated because of a lack of data. It is unlikely, however, that a fast flux in the neighborhood of 7.2×10^{17} nvt would result in appreciable embrittlement of as ductile an alloy as 80Ag-15In-5Cd. Thermal neutron irradiation of Ag-In-Cd alloys transmutes silver to cadmium, indium to tin while cadmium remains as an isotope. This implies that irradiation-induced composition changes could conceivably transform an originally ductile single phase Ag-In-Cd alloy into an embrittled two phase material. A method for estimating the composition change which would be induced in a Ag-In-Cd alloy by thermal neutron irradiation is given in reference 1. A calculation using this method has been made, and shows that a thermal flux of 2.4×10^{17} nvt will cause an immeasurable change in the composition of an 80Ag-15In-5Cd alloy. Composition changes, therefore, are not significant. Irradiation-induced precipitation in 80Ag-15In-5Cd is a possibility at the low temperature of NERVA operation (about 300 °R). This phenomenon is difficult to evaluate since the thermodynamically stable phases in this system are unknown at this temperature. It is unlikely, even if the single phase is unstable at the operating temperature, however, that a fast flux of only

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7.4×10^{17} nvt would result in enough second phase precipitation to seriously embrittle so ductile an alloy as 80Ag-15In-5Cd. This material is, therefore, judged adequate for cluster plate use from an irradiation damage standpoint. Arguments similar to those presented for 80Ag-15In-5Cd alloy can be put forth for the Ag-In-Cd-Sn alloy, and irradiation induced property alterations are unlikely.

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CONCLUSIONS

- 1) The yield strength of the annealed 80Ag-15In-5Cd test material was found to be much higher than that shown in the literature. This high yield strength is attributed to differences in fabrication and grain size.
- 2) Cold work provides a major increase in the strength of 80Ag-15In-5Cd. A room-temperature yield strength in the neighborhood of 65,000 psi can be obtained with approximately 50% cold reduction in thickness as opposed to 10 - 20,000 psi for annealed material. Adequate ductility is also displayed in cold worked material. Material in this condition rapidly recrystallizes at temperatures in the vicinity of 835 °R, but shows no recrystallization after 50 hours at 760 °R.
- 3) The strength level achieved by the tin-containing alloy in this investigation can be approached by annealed 80Ag-15In-5Cd. Because of this, the tin alloy does not appear to hold any definite advantages over 80Ag-15In-5Cd.
- 4) The strength of the two silver-base alloys drops off appreciably at temperatures above 800 °R.
- 5) The strength and ductility of 1% boron stainless remain high over the temperature range 140 to 1500 °R. The alloy displays some degree of notch sensitivity over this temperature range. Its strength, however, is so high (especially at low temperatures) that a stress concentration problem during reactor operation is unlikely. The alloy also displays an unusual betterment of notch properties at temperatures below room temperature. Of the alloys evaluated the 1% boron stainless displays the best combination of mechanical properties.
- 6) The mechanical properties of 80Ag-15In-5Cd and 1% B¹⁰ containing stainless steel will not be deleteriously affected by irradiation damage resulting from a 20-minute NERVA test.

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- 1) Neutron Absorber Materials for Reactor Control, Edited by D. Kermit Anderson and J. S. Theilacker (1962)
- 2) The Effect of Nuclear Radiation on Structural Metals, DMIC Report 166, September 15, 1961, Battelle Memorial Institute
- 3) Memo No: RA-553, D. McCutchen, WANL Reactor Analysis
- 4) Radiation Damage Studies of Boron Stainless Steel, Final Report, AEC Research and Development Report IDO - 16502, W. O. Shaffuit, Phillips Petroleum Company, Atomic Energy Division, 28 January (1959)

TABLE I
FABRICATING PROCEDURE USED TO PRODUCE WROUGHT PLATES
FROM STATICALLY CAST Ag-In-Cd and Ag-In-Cd-Sn

Ag-In-Cd		Ag-In-Cd-Sn	
Step	Processing	Step	Processing
Cast:	1-1/2" x 10" x 20"	Cast:	1-1/8" x 5" x ?
Roll (Cold)	1.5 to .650" 11 passes		
Annealed in Exogas:	1000 °F - 30 minutes	Condition:	.750 x 5" x ?
Flatten	--	Soak:	1000 °F - 12 hours
Overhaul (On Miller)	.030" off each face	Inspect & Condition	--
Roll (Cold)	.590 to .300 7 passes	Anneal:	950 °F - 1 hour
Trim Ends	--	Roll (Hot)	.750 to .440 4 passes
Anneal in Exogas	1000 °F - 30 minutes	Anneal	950 °F - 1 hour
Pickel	15% H ₂ SO ₄ (warm)	Roll (Hot)	.440 to .260 4 passes
Roll (Cold)	.300 to .150 6 passes	Anneal	950 °F - 1 hour
Flatten	--	Roll (Hot)	.260 to .158
Shear 2 pieces	3-1/2 x 20	Roll (Cold)	.150
Anneal 1 piece	752 °F - 1 hour	Anneal	900 °F - 1/2 hour

TABLE II
CHEMISTRY ON SILVER AND STAINLESS STEEL BASE ALLOYS

Element	Ag-In-Cd		Ag-In-Cd-Sn		Boron-Stainless
	Specified	Actual	Specified	Actual	Actual
Ag	80 ± .25	79.89	75.45 ± .25	75.69	--
Cd	5 ± .25	5.17	8.65 ± .25	8.43	--
In	15 ± .25	14.89	11.45 ± .25	11.40	--
Sn	--	< .001	4.45 ± .25	4.35	--
Cu	--	.08	--	< .05	.07
Zn	--	< .001	--	--	--
Fe	--	< .001	--	--	remainder
Mg	--	< .001	--	traces	--
Pb	--	< .001	--	traces	--
Bi	--	< .001	--	--	--
Mn	--	Not detected	--	--	1.76
Ni	--	Not detected	--	--	13.55
Si	--	Not detected	--	--	.61
C	--	--	--	--	.037
P	--	--	--	--	.012
S	--	--	--	--	.008
Cr	--	--	--	--	18.50
Mo	--	--	--	--	.12
B	--	--	--	--	1.06

TABLE III *

ROOM TEMPERATURE TENSILE PROPERTIES OF 80Ag-15In-5Cd CONTROL ROD EXTRUSIONS

Specimen Condition	Yield Strength (0.2% psi)	Ultimate Strength (psi)	Reduction In Area (%)	Elongation (%)	Hardness (DPH)	ASTM Grain Size
Extruded 1000 °F	14,000	46,000	56	52	95.8	8
Extruded 900 °F	15,200	45,000	52	44	99.3	8
Extruded 1100 °F	10,800	43,750	52	58	84.1	8
Extruded & annealed 752°F (1 hr)	13,900	45,600	60	57	89.0	6-8
Extruded 1100 °F	15,600	45,600	58	53	70.0	4-6
Extruded & annealed 752°F (1 hr)	11,800	44,200	46	52	67.0	4-6
Extruded & annealed 752°F (1 hr)	11,000	42,900	55	51	78.5	4-8
Extruded & annealed 932°F (1 hr)	7,700	38,600	60	61	67.8	1-3

*Reference 1

TABLE IV*
ROOM TEMPERATURE TENSILE PROPERTIES AND ELECTRON-ATOM RATIO
OF ANNEALED Ag-In-Cd-Sn ALLOYS

Alloy No.	Composition				Yield Strength (0.2%,psi)	Ultimate Strength (psi)	Elongation (% in 2")	Reduction of Area (%)	e/a
	Ag	In	Cd	Sn					
1	70.4	7.6	20.1	2.0	9,300	37,600	59	75	1.396
2	75.2	12.0	10.0	2.8	21,400	48,600	42	49	1.402
3	75.5	12.1	9.0	3.4	23,700	50,000	41	45	1.410
4	76.1	12.0	8.0	3.9	33,500	55,000	31	40	1.417
5	74.7	11.0	9.5	4.8	33,900	53,100	34	39	1.435
6	75.6	8.4	10.3	5.7	34,500	51,000	19	22	1.507

*Reference 1

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FIGURE 1

TENSILE SPECIMEN USED TO EVALUATE THE
 MECHANICAL PROPERTIES OF THE SILVER BASE ALLOYS

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FIGURE 2

TENSILE PROPERTIES OF ANNEALED 80 AG - 15 IN - 5 CD

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ANNEALED $80A_g-15In-5Cd$

100X
ASTM GRAIN SIZE 7 - 8
DPH (10K_g) 67

ANNEALED $80A_g-15In-5Cd$

200X

50% COLD WORKED $80A_g-15In-5Cd$

200X

$75.5A_g-8.6Cd-11.5In-4.45Sn$

500X

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FIGURE 3 MICROSTRUCTURES OF SILVER BASE ALLOYS

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FIGURE 4
HARDNESS VS TEMPERATURE FOR 1/2 HOUR ANNEAL
OF 50% COLD WORKED 80 AG - 15 IN - 5 CD

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FIGURE 5

TENSILE PROPERTIES OF 50% COLD WORKED 80 AG - 15 IN - 5 CD

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FIGURE 6
EFFECT OF TIME AT TEMPERATURE ON THE
HARDNESS OF 50% COLD WORKED 80 AG - 15 IN - 5 C D

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FIGURE 7

TENSILE PROPERTIES OF 75.5 AG - 11.5 IN - 8.6 CD - 4.4 SN

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FIGURE 8

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TENSILE SPECIMENS USED TO EVALUATE THE 1% BORON-STAINLESS ALLOY

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FIGURE 9
TENSILE PROPERTIES OF 1% BORON - STAINLESS

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FIGURE 10

NOTCHED TO UNNOTCHED TENSILE STRENGTH RATIO
OF 1% BORON STAINLESS

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FIGURE 11

PHOTOMICROGRAPHS OF 1% BORON-STAINLESS

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FIGURE 12 MECHANICAL PROPERTIES OF IRRADIATED 18-8 STAINLESS STEEL
CONTAINING 1% TOTAL BORON. (93% ENRICHED IN B^{10}) REF. 4

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