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JOINT REPORT OF METALLURGY AND PHYSICS DIVISIONS

INTERIM REPORT ON EFFECTS OF REACTOR RADIATIONS ON METALS

D. S. BILLINGTON

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Joint Report of Metallurgy and Physics Divisions

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D. S. Billington

Date Issued: OCT 27 1948

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I INTRODUCTION

The work described in this report is the Metallurgy Division contribution to the solid state program of the Physics Division. The objectives of this part of the program are as follows:

(1) To study the effect of reactor radiation on the mechanical and related properties of a wide variety of metals and alloys.

(2) To study the effect of reactor radiation on specific materials which are of considerable interest to such groups as the Power Pile Division, such as stainless steels.

(3) To study the effect of reactor radiation on specific metallurgical processes such as precipitation hardening, diffusion, grain growth, stress strain relationships, under variable conditions of grain size, prior cold work and heat treatment and microstructure.

(4) To study the effect of isolated nuclear particles on the behavior of metals and alloys, e.g., α-particles, x-rays, fast neutrons, slow neutrons.

(5) Devise suitable treatments for counteracting radiation effects if they appear to be harmful; e.g., determination of time and temperature relationship for annealing out observed effects.

(6) Develop suitable metallographic techniques for the remote preparation of samples for microscopic examination, and suitable means for observation of the microstructure, either directly or by replica techniques.

(7) Develop apparatus for the performance of mechanical tests remotely.

The work recorded below describes some progress toward the accomplishment of objectives 1, 2, 3, 6 and 7.

The principal reason for making a report at this time is to make a matter of record such data as have been accumulated up to the present time

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It is not felt that a detailed analysis of the effect of reactor radiation on the mechanical properties of metals and alloys can be made at this stage, consequently the data presented below will in general be augmented only by a few pertinent remarks on the observations, with no attempt being made to discuss mechanisms, etc. However, the accumulation of further data in the near future on stainless steels and copper beryllium should warrant a more detailed discussion of these two interesting alloys.

The work reported below was done with the active collaboration of the following persons:

Dr. S. Siegel	General guidance on all phases of the work
B. J. Johnson	Hardness measurements
M. J. Valentine	Electrical Resistivity measurements Dimensional changes Modulus and damping measurements
M. A. Bredig	X-ray diffraction studies
Dr. T. A. Read Lt. Comdr. Roth	Replica techniques
R. B. Goodrich	Electropolishing

Acknowledgement is also due to the following persons for the preparation, fabricating and metallography of certain alloys studied:

C. D. Smith

G. Adamson

Geo. Carlton

Acknowledgement is made to all the members of the Metallurgical Division for their active support.

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II MATERIALS STUDIED

The following list comprises the materials studied, or that are being irradiated preparatory to observation. The metals or alloys marked with a double asterisk have had measurements made on them, and are recorded in this report. The single asterisk denotes samples being irradiated, while those with no marking are in the process of preparation for irradiation.

TABLE 1

NOMINAL ANALYSIS (Weight percent)

MATERIAL	C	Cr	Ni	Mo	Cu	Cb	Mn	Si	Fe
Stainless Steel-Type 304 **	.08	18	9						
" " " 309 **	.20	25	13						
" " " 316 **	.10	17	12	2.5					
" " " 347 **	.10	18	10			1.00			
Monel **			67		30		1.0		1.4
Med. Carbon Hot Die Steel **	.40	5.		1.4	V-0.4			1.0	
High Carbon Hot Die Steel**	1.5			.25			.4	.8	
Nickel **			99.4						
Nickel High Purity **			99.99						
Iron High Purity **									99.9+
Grey Cast Iron *									
Austenitic Cast Iron *	3.0	2.5	16.0		6.5			1.6	
Nitrided Nitralloy(N-125)*	.25	1.15		.20			.5	.25	Al -1.2
Drill rod	1.0								
Heat Resisting Steel Casting *	.15	35							bal

[redacted]

TABLE 1 (Cont'd)

NOMINAL ANALYSIS

NON-FERROUS	Cu	Zn	Si	Al	Mn	Fe	Sn	Be	Mg
Yellow Brass **	65	35							
Silicon Bronze **	96		3		1				
Beryllium Copper **	97.7		.05	.05		.05		2.15	
Al Bronze *									
Tin Bronze *	90						10		
Copper **	CFHC								
Aluminum **				98+					
Beryllium **								99.8+	
Magnesium **									99.9+
Zinc **		99+							
Brass	93.57	6.43							
Brass	90.	10.							
Brass	84.13	15.87							
Brass	79.18	20.82							
Brass	74.18	25.82							
Brass	70.13	29.87							
Mg-Li Alloy	Li 10								90.

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III METHODS OF EXPOSURE TO REACTOR RADIATION

There are a number of different methods of exposure to reactor radiation possible. However, the results reported below were obtained under only two different conditions:

(1) Exposure in regular process channels at Hanford.

(2) Exposure in doughnut holes in ORNL reactor.

In the first case the temperature rise induced in the samples is not over 25°C. above cooling water temperature *, while in the latter case the temperature rise above inlet air temperature is not more than 20°C.

The above two methods of exposure give the best compromise of low temperature rise in specimen, plus fast flux.

Other methods of exposure, such as in a hollow uranium slug at Hanford, would give higher fast flux, but the temperature rise is estimated to be 100-150°C. This temperature rise is sufficient to preclude the study of many alloy systems which will undergo metallurgical transformations at this temperature.

In this preliminary part of the program it has been thought best to make all irradiations at low temperatures and then study the effect of subsequent annealing. Another approach is study effects when exposure is made at elevated temperatures. This part of the program is just being started.

Other methods of exposure such as Hanford Side Hole tubes, with and without water-cooling, are also contemplated for future work.

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* Provided good thermal contact is provided between the samples and the wall of the container. In the ORNL reactor good thermal contact is not required.

 [redacted]

IV OBSERVATIONS MADE

(a) Hardness

The hardness test has been used exclusively as a measure of the effect of Reactor radiation on mechanical properties. There are a number of reasons for this choice:

(1) The hardness tester used, the motor driven Rockwell machine, is easily adapted to remote operation.

(2) The size of sample necessary is small and thus is easier to handle, as the induced activity is lower.

(a) The space available for irradiation is small and it is important that as many samples as possible be exposed at one time.

(3) Less machining time and precision is required for sample preparation.

(4) Tensile tests are difficult to make remotely.

(5) In many materials the relation between hardness and tensile properties is well established.

(6) Space for tensile test equipment is limited at the present time. This does not mean that stress strain curves will not be obtained. On the contrary, when the behavior justifies it the stress strain relationships will be studied in great detail. A number of sub-size tensile bars are being irradiated at the present time.

(7) The hardness appears to be very sensitive to reactor radiation, at least under certain conditions to be described below.

A word of caution is necessary in regard to the hardness measurements. Because of the different Rockwell scales employed, it is not possible to compare accurately the results obtained using one scale with results on a different metal using a different scale. It should be noted also that the

 [redacted]

scales are more sensitive on the low end of the scale than on the high end. The hardness values reported represent the average of a minimum of four readings on each of at least two samples. All hardness samples were discs 3/8" diameter x 1/8" thick.

It is hoped in the near future to be able to use more appropriate hardness testers, such as the Vickers or Tukon machines, wherein the hardness measure in (Kg/mm^2) is independent of load and the range is unlimited.

(b) Electrical Resistivity

This test is easy to perform remotely, is sensitive to radiation effects and provides a means of giving an indication of the thermal conductivity, a property of much practical interest, but difficult to measure remotely. Sample dimensions for most specimens are 3/16" diameter x 2½" long.

(c) Modulus of Elasticity and Internal Damping

The measurements are easily performed on the resistivity samples, and the internal damping measurements are very sensitive to slight changes in internal structure.

(d) Dimensional Stability

This measurement is important from a reactor design standpoint, and in many cases is indicative of phase changes.

(e) X-ray Diffraction

These measurements have only been made on samples with short half life, but the observations should be valuable as a means of throwing light on the mechanism of changes induced by radiation. Changes in line width and intensity have been observed.

V DATA

(a) Stainless Steel

Stainless steel was chosen for experimentation, as it represents a class of material that is of interest in certain aspects of power reactor construction, as well as being a popular material useful because of strength, corrosion resistance and high temperature strength. It has a large capacity for work hardening, and will often show some phase transformation by cold work. Stainless steel samples have been exposed in the ORNL and Hanford Reactors. Some of the observations made on hardness changes are tabulated below:

TABLE II

Samples exposed for 3 weeks in Hollow Slug in Hole 1867, ORNL Reactor

Rockwell "B" Hardness Number (100 Kg load - 1/16" Ball)

Material	Condition	Before Irradiation	After Irradiation	Change
Type 316	hot rolled	81	84	+ 3
Type 309	"	84	85	+ 1
Type 347	slightly cold worked	90	93	+ 3

Samples similar to the above were heat treated by quenching in water from 1900°F. (Best treatment for corrosion resistance) followed by a three weeks irradiation in the ORNL Reactor. The following results were obtained:

		Control Samples	Irradiated Samples	
Type 316 - Quenched from 1900 F.		82.5	85	+2.5
Type 309	"	84	86	+ 2
Type 347	"	83.5	85.5	+ 2

The following samples were irradiated at Hanford in papoose slugs (sample slugs attached to one end of regular slugs in process channels). The estimated fast flux is of the order of 1×10^{13}.

TABLE III

Sample #	Condition	Days Exposed	Rockwell "B" Hardness Number (100 Kg load - 1/16" ball) Before Irradiation	After Irradiation	Change
Type 316	hot rolled	76	81.5	93.5	+ 12
Type 316	"	128	80.5	94.0	+ 13.5
Type 309	hot rolled	76	83.0	97.5	+ 14.5
" "	"	128	83.0	99.0	+ 16.0
Type 347	slightly cold worked	76	91.0	100	+ 9.
" "	"	128	90.5	101	+ 10.5

Discussion: The above stainless steel samples have shown an increase in hardness in both reactors. While only a slight increase is observed for the short exposures of ORNL, it is nevertheless real. The hardness changes induced by the Hanford irradiations are much more pronounced, as the samples show an appreciable increase in hardness. It appears that if stainless steel were left in a Reactor for an indefinite period, it might become brittle enough to rupture. (Stainless steel, if severely cold worked without immediate subsequent annealing, will often rupture.) This possibility should depend upon the following factors:

(1) The increase in hardness must be similar in nature to conventional cold working.

(2) The hardness must increase continually with exposure time.

(3) The temperature during exposure must be low.

It should be emphasized at this point that the stainless steels are capable of absorbing much more cold work than has been put into them by a six months exposure at Hanford. The increase in hardness would indicate the equivalent of 5% cold work, and the material could absorb probably 5, maybe 10 times this amount before the internal stresses set up would be serious. It should be noted also that conventional cold work practice develops directional properties in the metal and thus develops inhomogeneous internal stresses which lead to the rupture noted above. In the case of cold work induced by neutron bombardment it is felt that stresses are developed in a more uniform fashion, thus permitting a higher concentration of internal stress with less likelihood of rupture.

It is entirely possible, as noted under the copper beryllium section, that an equilibrium state is reached, at which the hardness change ceases. There is some indication of this in the observation that hardness obtained in the 128 day exposure was not much higher than the 76 day.

(b) Monel

Hardness samples of monel were exposed under the same conditions as the stainless steel. The observations are tabulated on the page following.

TABLE IV

Rockwell "B" Hardness Number (100 Kg load - 1/16" ball)

Sample Material	Treatment	Before Irradiation	After Irradiation	Change
(A) 3 weeks in ORNL Hole 1867				
Monel	Annealed	57	67	+ 10
(B) 3 weeks in ORNL Hole 1867				
		Rockwell "G" Hardness Number (150 Kg load - 1/16" ball)		
Monel	cold rolled	79	80	+ 1.0
"	"	79	80	+ 1.0
"	35 min. at 780-795°C	15	41	+ 26.0
(C) 3 months in HEW papoose slug in regular process channels, near pile center.				
		Rockwell "B" Hardness		
Monel	cold rolled	98	102	+ 4
"	"	98	101	+ 3
(D) 6 months in HEW				
Monel	cold rolled	98	102	+ 4
"	"	99	103	+ 4

As can be seen from the tabulation, the monel samples showed a hardness increase regardless of heat treatment. The Hanford exposures are particularly interesting as these samples show a hardness increase, even though they possess considerable previous cold work. However, as in the stainless steel samples, the 6 months exposure samples are not a great deal harder than the 3 months samples.

(c) Nickel

Specimens of nickel were exposed for 3 weeks in ORNL in both the cold rolled and annealed conditions:

TABLE V

Material	Condition	Rockwell "B" Hardness Number (100 Kg load - 1/16" ball) Before Irrad.	After Irrad.	Change
Nickel (not cobalt free)	cold rolled	92	92	0
" "	$\frac{1}{2}$ hr. at 745°C	45	60	+ 15

The electrical resistivity of similar material and also a sample of high purity nickel free from cobalt was measured before and after irradiation. The change observed was less than 0.3% (the accuracy of the method).

The cold rolled nickel samples did not show any change in length, but the annealed nickel samples showed a 0.03% increase in length.

(d) Steel

A series of steel specimens were irradiated in the ORNL Reactor for three weeks. The samples were heat treated to be relatively soft. Hardened samples are being irradiated at the present time.

TABLE VI

Rockwell "C" Hardness Number (150 Kg load-diamond penetrater)

Material	Condition	Before Irrad.	After Irrad.	Change
Med. carbon hot die steel	as received	31.2	31.2	0
"	30 min at 950°C; furnace cooled	46.3	46.8	+ 0.5
High carbon hot die steel	as received	28.1	29.4	+ 1.3
	30 min at 950°C; furnace cooled	31.7	33.8	+ 2.1
SAE 4340	as received	37.1	38.2	+ 1.1
"	30 min at 950°C; furnace cooled	34.5	36.5	+ 2.0

These results are interesting because this material, though in the softened condition, is still much harder than most other metals studied; yet there is an increase in hardness. The small change in hardness should not lead one to believe that the change is less. It is difficult to convert from one scale to another without a great deal of duplicate measurements, and where the range in hardness is too great, it is virtually impossible. Much of the confusion about hardness measurements will be removed when the space and facilities permit the use of a Vickers or Tukon hardness tester.

(a) Copper Beryllium Alloys

This alloy was chosen for study because it furnishes an excellent example of a precipitation-hardening alloy, and much information obtained on this system would be applicable to other alloys exhibiting this reaction.

The basis of most precipitation or age-hardening alloys is the fact that the solute metal shows increasing solubility in the solvent metal with increasing temperature. The solubility of beryllium in copper increases from approximately 0.5% at room temperature to more than 2.5% by weight at 875°C. This excess beryllium may be retained in solution at room temperature by rapid quenching in cold water. No hardening will take place below 100°C. This non-equilibrium condition is designated as the solution annealed state. The alloy is readily fabricated or wrought in this form. To obtain maximum mechanical properties in this alloy, it is heat treated by reheating the alloy in the temperature range of 250-350°C. for a few minutes to several hours, depending on the temperature. If the solution annealed condition is followed by cold work, such as forging, rolling, drawing or swaging, the rate of hardening during heat treatment is increased; the requisite hardening temperature is lower, and the final mechanical properties obtained better than without cold work. This state of being reheated is denoted as the heat treated condition. If the temperature of heat treatment is too high, in the range 400-500°C, the strength rapidly goes through a maximum, followed by a subsequent decrease; the electrical conductivity increases. With long continued treatment, the mechanical properties approach those of the solution annealed condition.

The effect of reactor radiation on this system was studied as a function of the state of heat treatment, that is, alloys were exposed in the solution annealed state, the heat treated state, the over aged state and the solution annealed, plus cold worked state.

Samples in various states of heat treatment were exposed for 3 weeks in doughnut hole 1867 in the ORNL Reactor and hardness measurements made on the irradiated samples, with the following results:

TABLE VI

Rockwell Hardness Number (150 Kg load - 1/16" ball)

Sample Number	Condition	Initial	Final	Change
X-1-2	Solution annealed ($\frac{1}{2}$ hr. 800°C) Plus 50% red. in area by rolling	87.0	86.7	-0.3
X-7-8	Solution anneal ($\frac{1}{2}$ hr. at 750°C)	14.0	44.0	+ 30.0
Heat treated				
X-15	Treatment of X-1-2 plus 16 mins at 290-303°C	101.8	101.2	- 0.6
X-11	Treatment of X-1-2 plus 30 mins at 303-310°C	102.4	100.4	- 2.0
X-13	Treatment of X-1-2 plus 60 mins at 304-311°C	96.0	95.3	- 0.7
X-9	Treatment of X-1-2 plus 180 mins at 298-303°C	93.9	93.3	- 0.6
Over-aged				
X-1B	Treatment of X-1-2 plus 55 mins at 496°C	65.	67.5	+ 2.5
X-B	Treatment of X-1-2 plus $\frac{1}{2}$ hr at 750°C, W.Q. plus 10 hrs at 498°C	57.	71.	+ 14.
X-19	Treatment of X-1-2 plus 10 hrs at 498°C	55.	71.	+ 16.

An examination of Table VI reveals that the solution annealed and over-aged condition appear to be most susceptible to reactor radiation, as these samples show appreciable increase in hardness number.

It should be noted also that despite the fact that the hardness increase is large, the hardness number reached is not high compared to the hardness obtainable by heat treatment. When samples being exposed at Hanford for long periods are available for examination, they should shed further light on the total increase possible.

The other interesting fact seen in Table VI is that samples possessing considerable original hardness do not show any increase in hardness, but on the contrary give some evidence that a softening process may be taking place. The change in sample X-1-2 is not significant in itself as the values are within experimental error, but the fact that not only this sample, but all of the heat treated samples, show a similar slight decrease may be indicative of a real softening. Here again long exposures at Hanford may indicate the true trend. Data on the Hanford exposed samples will be obtained within the next three months.

In addition to the above hardness measurements, electrical resistance measurements were made on samples of similar heat treatment, though the sample size and shape were necessarily different. Table VII shows the results obtained on samples exposed for three weeks in hole 1867 ORNL Reactor.

TABLE VII

Sample Number	Treatment	Original Resistance Corrected to 20°C x 10 ohm	Resistance after Irradiation Corrected 20 X-10	% Change
X-4	Solution anneal + 50% cold work	229.4	233.2	+ 1.7
X-5	Same as X-4 plus 1 hr at 300°C	179.6	181.7	+ 1.2
X-7	Solution anneal only	213.8	240.1	+12.3
X-16	Over-aged 1 hr at 500°C	130.0	139.3	+ 7.2
X-14	Over-aged 10 hr at 500°C	123.1	130.0	+ 5.6

A comparison of the data in Tables VI and VII show that the samples that had a large increase in hardness are also the ones that had a large increase in electrical resistivity. However, the samples that showed a slight decrease in hardness do not show a decrease in electrical resistance, but show a substantial increase. The increase in resistance of the hardened samples should indicate an increase in hardness.

S. Siegel has measured the resistance of several copper beryllium samples irradiated at Hanford, and has found a nearly uniform increase in resistance of approximately 10%, regardless of heat treatment. Inasmuch as sample X-7 shows roughly a 10% increase after a short exposure at ORNL, it is suggested that an equilibrium value of resistance is soon established in the more susceptible samples. Whether this will be seen for hardness numbers remains to be seen.

The resistance measurements on samples X-4, X-5 and X-7 have been repeated twice after waiting period of several weeks, in order to learn if there is any change in resistance as a function of time. No significant change was observed. See Table VIII.

TABLE VIII

Resistivity at 20°C

Sample No.	3 wks. after Radiation	6 wks	9 wks
X-4	10.9	10.8	10.9
X-5	8.5	8.5	8.4
X-7	11.2	11.2	11.3
X-8 (Control	10.0		10.1

The maximum temperature noted in hole 1867 has been 52°C. In order to be certain that long times at low temperatures had no effect on the hardness, control samples in the solution annealed state were heat treated for 3 weeks at 65°C. No increase in hardness was observed.

Dynamic modulus and internal damping measurements were also made on the above irradiated samples X-4, X-5 and X-7. The results are as follows:

TABLE IX

Sample No.	Modulus			Internal Friction		
	Before Irradiation	After Irradiation	% Change	Before	After	% Change
X-4	15.4	15.8	+ 2.6	22.9	7.3	- 68.1
X-5	17.3	17.4	+ 0.6	4.1	3.9	- 4.9
X-7	18.1	18.4	+ 1.7	16.5	17.2	+ 4.2

While the moduli of all three samples show an increase due to irradiation, the internal damping shows a decrease in two of the three samples. The tremendous decrease in sample X-4 is very surprising, as it suggests a heating mechanism rather than a distortion of the internal structure of the metal. It is possible that the small temperature rise during exposure is responsible for this change.

X-ray diffraction studies were made on several of the hardness samples that had been irradiated. No evidence of precipitation was noted in the irradiated solution annealed samples or the solution annealed plus 50% cold work samples, either before or after irradiation, but was readily observable in the heat treated and overaged samples before and after irradiation. In the course of the above observations it was noted that there was additional broadening of the diffraction lines in the samples, originally either cold worked or heat treated after being irradiated, but not in the samples that had been solution annealed or overaged. That is, the samples that showed least change in hardness and electrical resistivity showed the greatest degree of line broadening. It is not certain at the moment whether this is a real effect or not, as it is felt that the surface condition of the samples may be giving misleading results. Another series of samples, especially prepared for diffraction studies, is being irradiated so that the above observations may be checked.

CONCLUSIONS

The data obtained above show that:

(1) Reactor radiation has a definite effect on the mechanical and physical properties of copper beryllium alloys, even though the exposure is short. The nvt for most of the above sample exposures was of the order of 1×10^{17}.

(2) The data also indicate that the softer the original material, the more pronounced the effect.

It might be mentioned here that the conclusion under (2) is not inconsistent with Andrade's[1] observations on the effect of prior cold work on the resulting effect of α-particle bombardment on the creep rate of certain metals; that is,

(1) Andrade, Nature, Vol. 156, p. 113, (1945)

he noted that α-particle bombardment increased the creep rate more when the material was soft than when cold worked. Though the relation between the two experiments is uncertain, the similarity in observation is interesting.

OTHER COPPER ALLOYS

Since it had been observed that metals in the soft condition showed a greater hardness number increase than in the cold worked state, it was considered of interest to investigate this behavior in several copper base alloys. Several copper alloys were therefore irradiated in both the cold worked and annealed condition with the following results:

TABLE X

Exposure: 3 weeks in Hole 1867

Rockwell "F" Scale - 60 Kg Load - 1/16" Ball

Material	Condition	Before Radiation	After Radiation	Change
Copper	Cold Rolled	93.3	93.5	+ 0.2
"	1 Hr at 640°C.	35.	61.	+16.
Yellow Brass	Cold Rolled	100.5	101.5	+ 1.0
"	1 Hr at 750°C.	64.	71.	+ 7.0
Silicon Bronze	Cold Rolled	108.	107.5	- 0.5
"	1 Hr at 750°C.	65.	74.	+ 9.0

The above data bear out results obtained on other materials, that the softer the condition before irradiation the more pronounced the increase in hardness due to radiation.

The electrical resistivity of an oxygen free high conductivity sample of copper showed a 1.7% increase. Note that copper showed a change in hardness and electrical resistivity, while nickel showed only a change in hardness.

1. DIMENSIONAL CHANGES INDUCED BY REACTOR RADIATION

All samples were exposed three weeks in hole 1867 ORNL. The samples used were $2\frac{1}{2}$ inches long by 3/16 inch diameter. The method is not good to better than 2 parts in 25,000 or 0.01%.

TABLE XI

Material	Condition	Original Length	Final Length	Difference X 10^4	% Change
Nickel	Cold Rolled	2.5020	2.5020	0	0
"	Annealed	2.4648	2.4655	+ 7.	+ 0.03
Copper Beryllium	Cold Worked	2.5137	2.5139	+ 2	+ 0.01
"	Heat Treated	2.4978	2.4977	-1	- 0.005
"	Solution Annealed	2.5104	2.5085	- 19	- 0.08
S.S. Type 347	Annealed or Hot Rolled	2.5090	2.5091	+ 1	+ 0.005
SS Type 304	"	2.5120	2.5123	+ 3	+ 0.01

It is felt that the results on only two of the above samples are significant, the annealed nickel and the solution annealed copper beryllium specimens. The contraction noted in the case of the copper beryllium is consistent with the thought that precipitation is taking place. (This was not detectable by X-ray diffraction). The expansion in the case of the annealed nickel may indicate internal distortion.

2. THE ELECTRICAL RESISTIVITY OF SOME HIGH PURITY METALS

Samples of high purity copper, nickel, iron and zinc were examined for change of resistance upon short time irradiation in ORNL Reactor (3 weeks in hole 1867). The results obtained are tabulated below:

TABLE XII

Material	Condition	Resistance (20°C) (arb. units) Before Irradiation	After Irradiation	% Change
Copper OFHC	Annealed 2 hrs 500°C	34.66	35.26	+ 1.7
Nickel (co.free)	" 1 hr 700°C	197.7	197.8	+ 0.05
Iron (co.free)	" " "	201.8	202.4	+ 0.3
Zinc (High Purity)	" 1 hr 100°C	125.9	125.6	- 0.2

The method of measurement is good to only 0.3%, thus only the change noted in the copper is significant.

Samples of project beryllium, high purity magnesium and 2 S aluminum were exposed in the ORNL reactor for three weeks, with little or no change as a result of irradiation, as an examination of the tabulation below will show on the beryllium and magnesium. Where a positive increase is shown it is of such small magnitude as to be insignificant compared with other ferrous and non-ferrous metals and alloys. This observation is consistent with the lack of change noted in other properties by other workers.

Material	Condition	Rockwell "E" Hardness Number (100 Kg load 1/8" ball) Before Irradiation	After Irradiation	Change
Magnesium H.P.	As received	42.0	42.0	0
"	30 min at 310°C	39.0	39.0	0
Beryllium	As received	89.5	90.7	+ 0.8
"	1 hr at 950°C	87.5	88.7	+ 1.2
Aluminum	As received	48.0	48.9	+ 0.9
"	30 min at 310°C	29.0	29.7	+ 0.7

CONCLUSIONS:

It is evident from the above data that reactor radiation does have a definite effect on the hardness of a great many metals. Physical properties, such as electrical conductivity, are also effected but not always consistent with hardness changes.

The changes noted in general are of small magnitude and certainly are not to be considered harmful effects. It is noteworthy that the exposures in many cases were very short and the nvt of the order of 1×10^{17}. The Hanford exposures were

of the order of 1×10^{20}, and as is to be expected, these specimens showed a greater amount of change in properties. It is not known at the present time whether the change is continuous with exposure. There seems to be a slight amount of evidence that the effect may reach a saturation value. It appears worthwhile for the benefit of high flux reactor design to obtain long exposures at the highest possible flux; also exposures should be made at elevated temperatures.

The degree of change induced appears to vary from metal to metal, with little or no change being noted in the light metals, such as beryllium, magnesium and aluminum. The effect in pure metals appears to be less than in alloys.

The heat treatment prior to irradiation appears to have an effect on the change accompanying irradiation. This is particularly true in the case of the copper beryllium alloy.

It is also noted that such metals as aluminum, beryllium, magnesium and zinc which generally showed little or no irradiation effects all have thermal absorption cross sections less than 1×10^{-24} cm^2, while such metals or alloys as Fe, Ni and Cu that do show radiation effects all have thermal absorption cross sections approximately ten times as great. This suggests the need for study of radiation effects in the absence of fast neutrons, including metals with very large absorption cross sections. If absorption cross section (thermal) is important, then a metal like silver should show a big effect compared to copper. A superficial examination of fast absorption and scattering cross sections showed no such obvious correlation as noted above.

Metallographic Techniques

A. Electrolytic Polish and Etch

The problem of examining the microstructures of irradiated samples that have such a high activity that conventional methods are impossible, has been studied.

A certain amount of experimental work has been done with the object in mind of accomplishing the following:

(1) Prepare for metallographic examination by remote methods, samples of radioactive metals or alloys. This may be of importance where portions of a reactor assembly or similar object are to be examined microscopically but no preparation for examination has been made prior to exposure of the material to reactor radiation.

The thought that has been pursued here is to develop suitable electro-polishing baths that will produce an etched surface suitable for microscopic examination or a bath that will produce a well-polished surface that subsequently may be etched either electrolytically or chemically.

This problem is particularly acute at present for samples of aluminum-uranium alloys that have been irradiated at Hanford and thus most attention has been focused on baths suitable for the polishing and etching of these alloys.

A number of baths were investigated. The most promising one found by R. B. Goodrich had the following composition:

KOH	1%
K_2CO_3	2.5%
$Na_3PO_4 \cdot 12\ H_2O$	2.5%
Water	balance

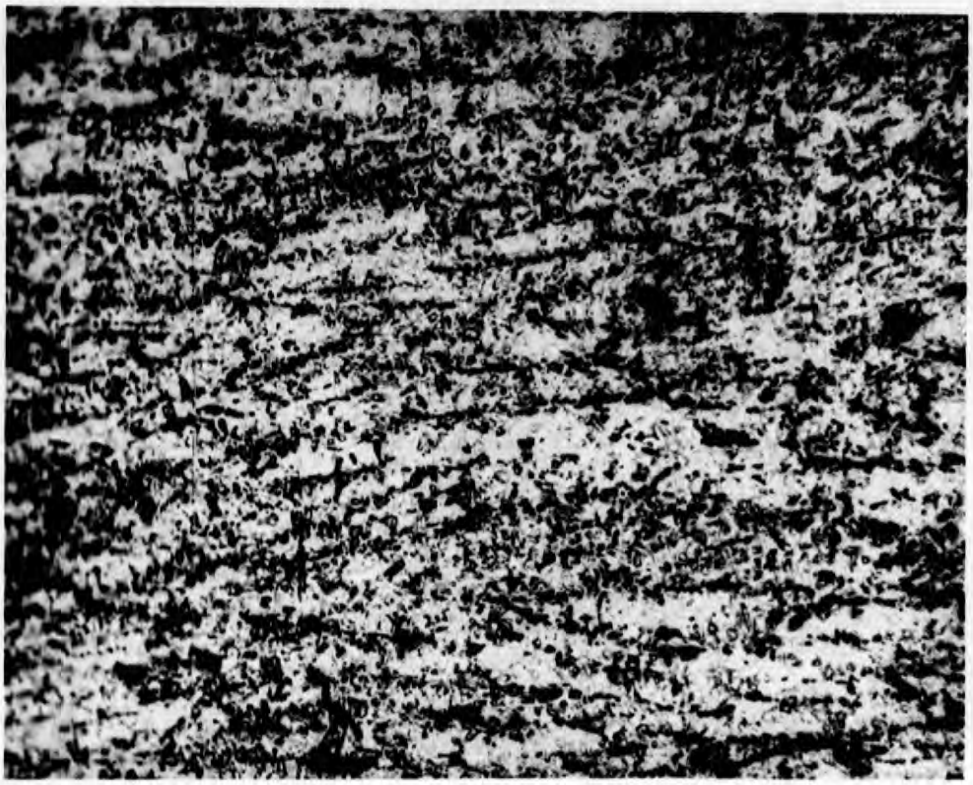
Temperature	80 - 100°C
Time	10 - 30 min
Voltage	5 - 15 volts
Current density	approx. 0.2 amp/cm^2
Mild mechanical stirring	

The bath works very well if a preliminary finish with 3/0 emery paper is placed on the sample. Polishing is fair without any prior polish or grinding. A photomicrograph is shown below. It is believed that conventional baths in use for such metals as copper will be perfectly satisfactory for polishing and etching of non-fissionable metals.

5% U-Al Alloy

250 x

$KOH-K_2CO_3$ Bath



B. Metallographic Replica Technique

The discoloration of glass and the deterioration of lens cements by high level radiation makes undesirable the direct viewing by periscopic microscope of radioactive metallographic samples. Therefore, it is desirable to develop a technique of making a replica of such surfaces.

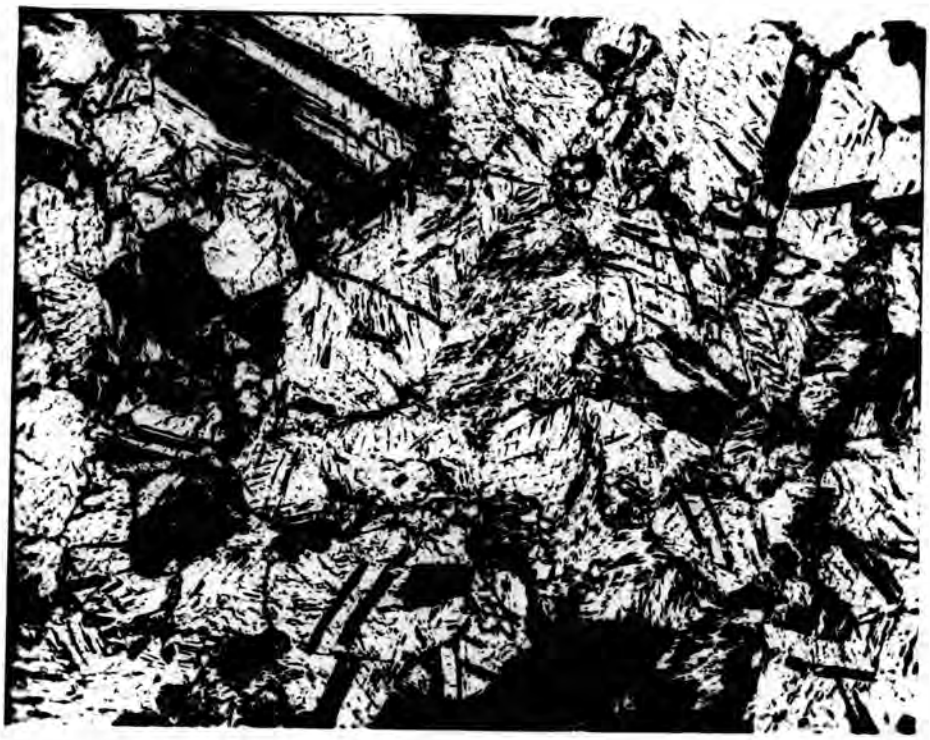
There are now in common use several methods of making metallographic replicas. They involve using plastics which must be softened before applying to the metal surface. There are two strong objections to use of such a method for radioactive samples. The radioactivity is known to discolor and decompose plastics, and handling of liquid solvents and wet surfaces behind the hot laboratory screen is a time consuming and difficult operation.

It was decided to try a replica technique using indium as the replica material because of its great plasticity. The process in its present stage of development involves preparation of a replica blank by pressing an indium sample to a block of polystyrene softened with benzol. A polished steel block is used to apply the pressure and smooth the surface. After drying, this blank is pressed against the polished and etched metallographic sample. This is the only operation done behind the radiation shields. The reproductions of surfaces have been examined to 500 diameters with excellent results in some cases. The etching depth and type of etchant used seems to be important since Be-Cu and Al-U samples showed excellent results while a sample of stainless steel did not give good results.

The photomicrographs below show the original sample and the replica reproduced on indium metal. The replica is, of course, a mirror image of the original.

Further development of this method is required in the direction of improving the initial indium surfaces, improving the band between the indium and the polystyrene, and in studying the etchings best suited for each metal under examination.

Cold Worked Cu-Be Alloy
Dichromate Etch, 500 x



Original

Replica