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no. 44088

The Role of Crystal Structure on Irradiation Effects on Metals[†]

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[†]This work was presented as a thesis by D. Wruck to the Graduate College of the University of Illinois in partial fulfillment of the requirements for the degree of Master of Science in Metallurgical Engineering in February, 1954. This work was supported in part by the U.S.A.E.C. ✓

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Abstract

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The resistivity of Fe has been determined to increase more than that of Co and Ni when these metals are bombarded at -150° C by 12 Mev deuterons. The same effect was observed for Fe and Ni by neutron irradiation at room temperature. This result may be interpreted as indicating that Fe is affected to a greater extent than either Co or Ni by heavy particle bombardment, though other interpretations may also be made. Annealing of the cyclotron irradiated samples showed that a smaller fraction of the effect produced in Fe remained after a room temperature anneal than remained in Co and Ni.

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Introduction

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One of the facts which seems clear from experimental studies of irradiation of metals is that different metals are affected by different amounts. It is of interest to determine what properties are responsible for this difference. Two properties which have previously been investigated in this regard are the effect of atomic number, Z , and the effect of the binding energy of the lattice. The first of these seems to be well established by measurements on Cu, Ag and Au, metals which are quite similar except for atomic number. Marx, Cooper and Henderson observed for these metals, upon bombardment at about -130°C , a change in resistivity which was larger the higher the atomic number 1/. This effect has also been observed at 12°K for these metals by Cooper, Koehler, and Marx 2/. If the damage (say in terms of interstitial-vacancy, I-V, pairs) is approximately equal for equal resistivity changes in these metals, as seems reasonable, then these experiments may be readily interpreted as showing an increasing effect for increasing Z . The effect of binding energy is not as clear cut with regard to degree of damage produced during bombardment. Although larger resistivity changes have been observed in Ni and Ta than in Cu, Ag and Au, 1/ because of differences in other properties (electronic structure of the metals, for example) one cannot express these resistivity changes in terms of actual differences in amount of damage. An effect was observed upon annealing which was attributed to differences in binding energy.

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Another property of a metal which may play a role in determining the degree of damage is the crystal structure. This property might well be a factor both in affecting the number of displacements occurring during bombardment and the amount of annealing taking place at a given temperature either during or after bombardment. Clearly, to make a valid comparison between metals of different structure type, one must choose metals which differ little from each other in all properties except this. Three metals which seem to satisfy these requirements quite well and at the same time allow comparison between the most common lattice types (for metals) are Fe, Co and Ni, respectively bcc, hcp (almost ideal) and fcc. Their atomic numbers are as close as is possible and their binding energies are about the same. They are all ferromagnetic (the importance of this feature is, of course, not known). Their electrical resistivities are not greatly different. This is of importance since this property was used in the present investigation to record the status of the damage. Some properties of these metals are listed in detail in Table I.

This paper reports on a series of measurements made on these three metals (called here the "primary group"). The results may be divided into two categories: (1) differences in damage produced by bombardment and (2) differences in annealing after bombardment. Two separate irradiations were made: (a) cyclotron irradiation with deuterons at low temperatures (-160°C to -100°C) and (b) neutron irradiation at ambient pile temperature.

Ti and V were investigated under cyclotron bombardment in an effort to provide substantiating information on another fcc and another bcc metal. Data obtained for these metals, while apparently correct, cannot at this

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time be used either to substantiate or refute the claims made for the primary group. This is so because one does not really know on what basis to compare them since their initial resistivities are greatly different from the others.

Metal	Crystal Type	Distance of Closest Approach	Z	at.wt	P20°C	T _m	Heats of Sublimation at R.T.
Iron	bcc	2.48A°	26	56	9.7μ _A cm	1540°C	94 Kcal/mol
Cobalt	hcp	2.50	27	59	6.2	1495	85
Nickel	fcc	2.49	28	59	6.8	1455	85
Titanium	hcp	2.95	22	48	~80	1820	100
Vanadium	bcc	2.63	23	51	~26	1735	85

Table I. Some physical properties of the metals bombarded.

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Experimental Procedure

A. Cyclotron bombardment

Foils of the metals were bombarded at low temperatures in the University of Illinois cyclotron. The experimental procedure was much the same as that used earlier in this laboratory 1/. The specimens were polycrystalline foils about 4 cm long, .2 cm wide and about .003 cm thick.* These foils were mounted for bombardment on an aluminum block connected to a liquid nitrogen container. The geometrical arrangement of the apparatus is shown in Figure 1. The deuteron beam entered the apparatus through a defining slit and passed through a .001" copper sheet before striking the foils. This copper sheet served to stop any heavy atoms which were carried along by the beam. That part of the beam not stopped in the foils was stopped by a thick block. An aluminum shield around the entire block served to collect any secondary electrons which might be produced. The flux of deuterons falling upon the samples was determined by measuring the charge collected by the foils and target block.

Since low temperatures were necessary, the entire assembly was designed to provide good heat transfer from the foils to the liquid nitrogen. The difficulty of making good thermal contact was increased by the necessity that electrical resistance measurements be made on them separately. This required that the foils be electrically insulated from the block at one end; a requirement that was accomplished by the use of thin mica insulating sheets between one end

*The purity of the foils was about as follows:

Fe ~ 99.95% pure, .04% O₂, not much metallic impurity

Co ~ 99.4% pure. Major impurities iron and nickel

Ni ~ 99.5% pure. impurities not known

V known to contain .14% C, .12% O₂, .11% N₂

Ti commercial purity.

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of the foils and the block. The foil temperatures obtained were not altogether satisfactory for all runs; it was found to be difficult to make the thermal contact equally good for all foils. In two of the runs, one or more of the foils got as warm as -120°C with the beam on. In the final run, however, all the metals in the primary group were below -145°C . Even with higher foil temperatures on some runs, all the data show the same effect as far as the effect of crystal structure is concerned.

The resistivities of the foils were determined from measurement of the IR drop produced in them by a known current. The gauge length (about 1.2 cm) was fixed by two .010" Cu wires spot-welded to the foils near the end of the irradiated section. The temperature of each foil was measured by means of a Cu-constantan thermocouple spot-welded to the back of the foil in the center of the irradiated section.

B. Pile Irradiation.

Twelve specimens of iron and twelve specimens of nickel were irradiated in the Brookhaven reactor*. The specimens were coarse-grain polycrystalline wires .030" in diameter and about 6 inches long. The large grain size was produced by first straining fine-grain wires about 1% and then annealing them for 7 days in vacuum at 850°C . The grain size of the iron wires was about $1/4"$ and that of the nickel wires about $1/32"$. Since both the iron and nickel wires were decarburized before this treatment, they were very soft.

The resistivity of the wires at 77°K was determined over a 2 inch length of the specimens again by measuring the IR drop produced when a known current

* Cobalt was not used in this measurement because the high radioactivity resulting from neutron irradiation would have made handling of the samples difficult.

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was passed through them. For measurement, the wires were mounted in a jig which was immersed directly into a bath of nitrogen. Resistivity determinations were made both before and after irradiation to determine the effect of bombardment. Further measurements were made after the specimens had been annealed at successively higher temperatures up to 500° C. Irradiation of the samples was carried out at 50° C for two months. The integrated neutron flux was 1.25×10^{19} neutrons/cm².*

* This pile bombardment actually had a two-fold purpose: (1) One of these was to compare the resistivity increase in iron and nickel attendant upon a given neutron irradiation. (2) The second was to see if any anelastic effects could be observed above room temperature in irradiated iron or nickel. Specifically, we thought there was a possibility of observing a relaxation peak of interstitial iron in the bcc iron lattice similar to that observed for other interstitial atoms in bcc lattices (for example interstitial C and N in bcc iron). Observations on nickel were expected to show no effect of this kind since this anelastic process does not occur for interstitial atoms in fcc lattices. No significant changes after irradiation were observed in the anelastic behavior of either iron or nickel in the region from 25° C to 600° C. One is thus lead to the conclusion that, if interstitials are produced by bombardment, (1) they are either too few in number to change the anelastic behavior significantly or (2) they move in iron at a temperature lower than room temperature.

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Results

A. Cyclotron Irradiation.

Bombardment.

A preliminary measurement of the increase in resistivity accompanying bombardment at low temperature of Fe, Ni and Co served to demonstrate that significant differences did exist. In this determination, Run I, the foil temperatures were not maintained as low as was believed desirable (-120°C during bombardment), so further measurements were carried out with lower beam currents and better foil-mounting techniques. The second measurement (Run II) was more successful, though the Ni foil again was rather warm (-120°C) because of an accident in foil mounting not discovered until the end of the run. The final run, (Run III), was quite successful from this point of view in that all foils were rather cold ($<-145^{\circ}\text{C}$). Quite possibly the limiting factor in Runs II and III (except for Ni in Run II) was the rate of heat transfer down the specimens to the cooling block.

The changes in resistivity which were measured in Run II for the four metals Fe, Co, Ni and Ti are plotted in Figure 2 as a function of integrated neutron flux. Ni and Co show rather small changes in resistivity compared to those observed for Fe and Ti. The data for Ti may be somewhat misleading when comparisons are made from this plot, for the initial resistivity of Ti was at least 6 times that of any of the other foils. If these data are plotted as the fractional change in resistivity, as in Figure 3, then the relative positions of Fe, Co and Ni are unchanged, but Ti also falls to a rather low level.

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Similar data, Run III, for the primary group together with data for V are shown in Figures 4 and 5. The behavior of Fe, Co and Ni is relatively the same as in Figures 2 and 3. V behaved somewhat as Ti did; it had a rather ^{high} absolute ^{because} change in resistivity, but a rather low fractional change of its high resistivity. (Though Figures 4 and 5 show but one curve for V, two foils were actually mounted and run. They behaved so similarly that data for but one are plotted.)

A summary of the data pertinent for comparison purposes is given in Table II, along with a piece of data for Ni taken from the paper of Marx, Cooper and Henderson. In this table are given (1) the values of initial resistivity of the foils at -180°C (this was the equilibrium value of temperature obtained during measurement with about 1 ampere of current flowing through them), (2) the highest temperatures reached by the foils during bombardment, and (3) the resistance change recorded at the end of each run expressed both in absolute value and fractional change.

With regard to Fe, Co and Ni, the chief conclusion which can be drawn from these data is that Fe undergoes a greater resistivity change upon bombardment than either Co or Ni (which behave in about the same way). The change is greater in Fe by a factor of from 3 to about 6 depending upon whether the comparison be made through changes in ρ or in $\Delta\rho/\rho_i$. Ti and V behave either more like Fe or like Co and Ni depending on the type of comparison.

The shape of the bombardment curves as a function of integrated deuteron flux is closely the same for all of these metals. In Figure 6 is plotted the fraction of total resistivity change vs. integrated flux for Run III. The final point for each metal is normalized to unity. This composite curve shows that, whatever is the phenomenon which causes the bombardment curve to bend

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over, it is constant (or changes similarly) in these four metals during the course of the bombardment.

Metal	Temperature of foils during bombardment	Initial resistivity at -180°C . ρ_i	$(\Delta\rho)_f$	$(\Delta\rho)_f$ ρ_i
Run I (Total flux 167×10^{15} deuts. / cm^2)				
Fe	-120°C	$2.635 \mu\Omega \text{ cm}$	$1.193 \mu\Omega \text{ cm}$	.453
Co	-120°C	2.803	.211	.075
Ni	-120°C	3.493	.430	.123
Run II (Total flux 67×10^{15} deuts. / cm^2)				
Fe	-150°C	1.647	.583	.354
Co	-145°C	2.167	.169	.078
Ni	-120°C	3.232	.177	.055
Ti	-100°C	20.96	1.45	.069
Run II (Total flux 97×10^{15} deuts. / cm^2)				
Fe	-160°C	2.25	1.218	.541
Co	-150°C	2.67	.283	.106
Ni	-145°C	4.16	.351	.084
V ₁	-135°C	17.96	.709	.039
V ₂	-135°C	16.49	.647	.039
Data of Marx et al ¹ (Total flux 114×10^{15} deuts. / cm^2)				
Ni	-133°C	3.123	.381	.122

	.541	
	.106	
	.084	
	.039	
	.039	

Table II. Resistivity data for 4 bombardments.

Annealing

At the conclusion of each bombardment, some annealing experiments were carried out. In Run I pulse anneals were made by using the Joule heat of an ac current to maintain the foils at a given temperature for an hour or more. An accidental loss of vacuum prevented the taking of annealing data for Run II except for a room temperature anneal.

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A "saw-tooth" type of anneal was carried out after Run III in which the foils were allowed to warm up to a given temperature as the entire apparatus warmed up after all the liquid nitrogen evaporated. The results of the "pulse" anneal and "saw-tooth" anneal were quite similar.

The results of the anneal for Run III are presented in Figure 7. The ordinates refer to resistivity measurements made (at -180°C) after the foils had warmed up to the temperature indicated. For convenience in comparing the metals, the curves are normalized. The rate of warm-up was about 20°C/hr except near room temperature, where it was much slower. In the region from -150°C to room temperature, Fe is seen to anneal somewhat more (fractionally) than do Co and Ni. The rather sudden drops at roughly -100°C for Co and Fe were observed in the annealing measurements of both Runs I and III (also both V foils showed closely the same drop in Run III). What process this corresponds to is unknown. A summary of data obtained after room temperature annealing is given in Table III. Here are given the final resistivity change after bombardment, $(\Delta\rho)_f$, the resistivity change still remaining (at -180°C) after room temperature anneal, $(\Delta\rho)_A$ and the fraction remaining after the room temperature anneal. The amount of annealing seems to increase from Ni to Co to Fe.

Some anneals above room temperature to 500°C were also attempted. They were not highly successful because of a large scatter of points. Within the spread of the measurements, there seemed to be no sharp temperature range in which the remaining resistivity disappeared.

B. Neutron Irradiation

Resistivity changes in Fe and Ni following neutron irradiation at 50°C showed the same trend as the cyclotron measurements. Table IV shows data for 6 of the specimens irradiated. The absolute change in resistivity was about 4 to 5 times greater for Fe than for Ni, the fractional change, more than double.

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Measurement	Metal	$(\Delta \rho)_f$ $\mu \Omega \text{ cm.}$	$(\Delta \rho)_A$ $\mu \Omega \text{ cm.}$	$1 - \frac{(\Delta \rho)_f - (\Delta \rho)_A}{(\Delta \rho)_f}$
Run I, 72 hrs. at RT	Iron	1.193	.268	.22
	Cobalt	.211	(.026)?	(.01)?
	Nickel	.430	.145	.34
Run II, 6 hrs. at RT	Iron	.583	.125	.21
	Cobalt	.169	.055	.33
	Nickel	.177	.074	.42
	Titanium	1.45	1.05	.72
Run III, 24 hrs. at RT	Iron	1.213	.179	.15
	Cobalt	.233	.079	.28
	Nickel	.351	.151	.43
	Vanadium I	.709	.320	.45
	Vanadium II	.647	.315	.49
Marx et al (Ref. 1)				
20 hrs. at RT	Nickel	.335	.151	.39
96			.136	.35
264			.120	.31

Table III Effect of annealing at room temperature on bombarded foils.

Metal	ρ_{initial}	ρ_{final}	$\Delta \rho$	$\Delta \rho / \rho_{\text{initial}}$
Iron 1	.7786 $\mu \Omega \text{ cm.}$	.8509 $\mu \Omega \text{ cm.}$	.0723 $\mu \Omega \text{ cm.}$	.093
2	.7686	.8509	.0823	.107
3	.7410	.8192	.0782	.106
Nickel 1	2.243	2.256	.013	.006
2	2.423	2.445	.022	.009
3	2.248	2.267	.019	.008

Table IV Effect on resistivity (at 77°K) of neutron bombardment of Iron and Nickel.

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this. Annealing measurements made on these wires were limited somewhat since two of the Fe wires broke in handling (the remainder were used in anelastic measurements). Again the annealing data obtained showed too much scatter to allow detailed analysis of the results.

Discussion

The cyclotron measurements show that Fe undergoes a much larger change in resistivity under low temperature deuteron bombardment than do Co and Ni. The data for room temperature irradiation of Fe and Ni also show the same effect for these two metals alone. In both instances, the effect is so large compared to the differences one gets in making duplicate measurements on the same metal, that there is small chance of its not being genuine. Interpretation of the observed effect seems not so clear-cut, however. One might suppose that either of these two extreme points of view might apply: (a) There are indeed more I-V pairs produced in Fe than in Ni and Co or (b) The same number of I-V pairs is produced in the three, but they cause a larger resistivity change in Fe than in the other two.

In trying to decide between these two (or some combination of them), one sees that a number of possibilities exist, even for these three quite similar metals. Some of these possible factors are intrinsic physical properties of the metals themselves and some are various unresolved features of radiation damage itself. The factors which will be considered here are (1) purity of the materials, (2) energy required to produce an I-V pair, (3) electronic structure of the metals, and (4) differences in annealing behavior. Unfortunately, none of these factors can be completely eliminated as a possible explanation for the effect observed.

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(1) The importance to radiation damage of the purity of the material being bombarded is not known, although a study has been made of resistivity changes accompanying bombardment of some alloys of Cu (of reasonably high alloy composition compared to normal purity of metals)^{3/}. These results, reported for resistivity changes following neutron irradiation around room temperature, vary so much between alloys that no generalities were drawn about the effect of added elements. The work does indicate that there exists the possibility that impurities may be important in pinning down either vacancies or interstitials. However, since any attempt to interpret the present results in terms of the purity of the materials is virtually pure speculation at present, this effect will not be considered further.

(2) There are numerous ways in which crystal structure could play a role in radiation damage. One of these is through its effect on the energy necessary to produce an I-V pair. It is easy to see that this could be the decisive factor in explaining the present experiments. Assume that the energy necessary to produce an I-V pair in Fe is less than that required in Co and Ni. Then, under similar bombardment, more pairs would be produced in Fe than in the other two by a given number of incident particles because more of the collisions (both of the primary and secondary nature) would involve a transfer of energy greater than this minimum. Then the greater resistivity change measured for Fe would simply reflect this larger number of I-V pairs produced.

To see if this conclusion is reasonable, one must examine the assumption that the threshold energy in Fe is less than that in Co and Ni. The argument favoring this point of view is the following: Fe, being bcc, has 8 nearest neighbors at a distance of about $2.5 \text{ \AA}$; Co and Ni, hcp and fcc respectively, have 12 nearest neighbors also at a distance of about $2.5 \text{ \AA}$. The potential energy barrier

over which a knocked-on atom must pass to be permanently displaced (even when projected in the most favorable direction), is then less in the bcc lattice than in the close-packed lattices because at the saddle point it passes close to a fewer number of atoms in the bcc lattice than in the close-packed lattices. In other words, for a knocked-on atom of a given energy, there is a larger solid angle of directions of projection which will result in permanent displacement in the bcc lattice over that in either the hcp or fcc lattices.

(3) The electronic structure of the metals may play a decisive role in that the same number of I-V pairs in each might cause quite different resistivity changes in the three. Hence, the different resistivity changes observed may not actually reflect the production of more damage in Fe than in Co and Ni. Two sources from which this electronic effect might stem are these: (a) The interstitials produced might be ionized to different degrees. In such a case they would certainly scatter electrons differently and hence would contribute different resistivity changes per defect. (b) The conduction electron band may have a different configuration.

There is little experimental evidence to decide whether either of these effects is important. One piece of information which may be of some significance in evaluating the second, is knowledge of the resistivity change occurring in these metals when impurities are added to them. Examination of published data shows that when a given metallic impurity is added to Fe and Ni*, the resistivity change occurring in Fe is usually somewhat greater than that occurring in Ni. For some impurities, the effect is about the same, for others, more than twice as great in Fe. Let an average factor somewhere between 1 and 2 be considered valid. If a factor of say, 2, is applied to the present results (there is no evidence to

*Not much data are available for Co.

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either support or refute the correctness of doing this), it still does not explain the difference of from 3 to 6 that is observed in the bombardment.

(4) Annealing during bombardment can arise from two sources, from thermally activated processes and from the incident particles. The details of annealing of close pairs seem obscure, but the rate of annealing of the widely separated defects is controlled by the rate at which one of them will diffuse through the lattice. Since this diffusion rate may depend markedly on the crystal type, it is reasonable to presume that the rate of annealing itself may be a function of crystal type. Unfortunately, for none of these metals is the heat of activation for motion of either defect known, although some heats of activation for self-diffusion in these metals are known.*

The annealing experiments reported in this paper can offer some information for evaluating the importance of annealing in the present discussion. After bombardment, not much thermally activated annealing is observed until the foil temperatures reach about -120°C , above which the annealing is rather rapid. It is interesting to note that for Fe (which shows the greatest resistivity change upon bombardment) the fractional annealing is greatest (Figure 7 and Table III). Hence, it appears that thermally activated annealing is not the cause of the difference in behavior upon bombardment, unless for Co and Ni annealing affects below -150°C are much more important than for iron. The curve shown in Figure 6 gives some evidence that this is not so.

A reasonable summary of these points is the following: (1) The effect of purity is not yet established. (2) The effect of crystal structure through its effect on the threshold energy for production of defects could be the important factor. (3) Different electronic structure of the metals could account

*For Fe and Co these are, respectively, 60 and 62 Kcal/mol.

for a part or all of the observed difference. (4) The effect of annealing cannot yet be fully evaluated.

A satisfactory interpretation of the experimental effects noted must also take into account the data obtained for V (bcc) and Ti (hcp). If crystal structure is an important factor in determining the relative amount of damage in different metals, then one would expect V to behave like Fe and Ti like Co and Ni. In comparing them with Fe, Ni and Co, one does not know whether to compare $(\Delta\rho)_f$ or $(\Delta\rho)_f/\rho_i$. Unfortunately, as an examination of Figures 2, 3, 4 and 5 shows, neither comparison is consistent for both. However, because of the extremely large values of ρ_i for V and Ti, it is possible that no comparison is a valid one.*

Acknowledgment is made of assistance and advice from the entire radiation damage group at the University of Illinois. The cyclotron bombardments were made possible by the cooperation of Professor Jentschke and his group. The neutron irradiation was carried out for the authors by Drs. Dienes and Fleeman of the Brookhaven National Laboratories.

*It is probably not yet decided whether Ti and V are intrinsically poor conductors or whether they have not yet been made pure enough to be good conductors.

Captions for Figures

- Figure 1. Cryostat and mounting block assembly.
- Figure 2. Resistivity change of foils in Run II.
- Figure 3. Fractional change in resistivity of foils in Run II.
- Figure 4. Resistivity change of foils in Run III.
- Figure 5. Fractional change in resistivity of foils in Run III.
- Figure 6. Resistivity changes in Run III normalized at 97×10^{15} deuterons/cm².
All experimental points for the metals fall randomly within the ranges indicated.
- Figure 7. Anneal to room temperature of foils in Run III.

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Figure 2.

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Run II

$$\frac{\Delta \rho}{\rho_i}$$

Fe

Co

Ni

Ti

$\times 10^{15}$

Deuterons / cm²

Figure 3.

Figure 4.

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Figure 6.

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FRACTION OF $(\Delta P)_f$

TEMPERATURE °C

Figure 7.

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