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A REVIEW OF ION SIMULATION OF HIGH-TEMPERATURE NEUTRON DAMAGE AND VOID FORMATION

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Introduction

The use of beams of charged particles to simulate high temperature neutron damage is only four years old. This technique was introduced in 1969 by Nelson and Mazey (1) and it was clearly aimed at obtaining information on the behavior of cladding material for fast reactors. Since that time, over 60 papers have been published in the open literature and at conferences (2-6) in addition to countless internal company reports. The nature of the studies have ranged from purely theoretical to quite empirical with some countries actually using the experimental programs to guide the design of multi-million dollar fast reactors.

It is difficult to gain the proper perspective of a field of research which is so young. Part of this difficulty stems from the fact that some of the important information is not being released because of commercial considerations, and part of it comes from the fact that many of the programs are still identifying experimental problems, let alone solving them. Nevertheless, tremendous progress has been made in a relatively short time and the future of this technique is indeed quite bright.

The object of this paper will be to sketch a brief historical description of how the field evolved and to summarize the various types of studies that have been performed. The bulk of the paper will be devoted to highlighting recent successes of neutron damage simulation. In addition to listing the advantages of this technique, we will attempt to be quite candid about its many limitations. Finally, a few of the more promising future studies will be explored.

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The Necessity and Validity of Such Studies

It was not long after the 1966 discovery that high temperature neutron irradiation of metals could cause significant swelling (7), that designers of fast breeder reactors realized the tremendous economic impact of that phenomena. The early expectation of the swelling in 316 stainless steel, the primary cladding material for the U.S. LMFBR program, is shown in Figure 1. This figure shows that swelling values of 2-15% were expected in the FFTF reactor and 7-60% may be possible in commercial LMFBR's. These values were put into persepctive by Huebotter and Bump (8) who stated that if 5% swelling must be accomodated in all the fast reactors built in the period 1970-2020, the present worth of a solution was $864,000,000. It was also stated that if the swelling amounted to 15%, then a 1970 present worth value was $5,600,000,000. Obviously, there was a sufficient economic incentive to solve the problem.

Unfortunately it appeared that it would take 5-10 years or more to obtain confirming data for Figure 1 if fast test reactors such as EBR-II and DFR were used. It was necessary to know the answer sooner than that.

Not very much was known about how voids formed in the 1966-1969 period. However, scientists have now shown that even though vacancies and interstitials are produced in equal numbers during irradiation, more interstitials than vacancies are absorbed by the dislocations in the solid. This imbalance leaves an excess of vacancies in the lattice which builds up to a critical supersaturation such that the vacancies finally precipitate into voids.

Many authors, too numerous to list here but referred to in (2-6) have developed theories to explain the swelling in reactor components. We will use the ideas developed by Bullough and coworkers (9-12). The main features of their theory are summarized in the equation 1 which applies after a threshold damage of Kt_o has been reached where K is the displacement rate in dpa sec^{-1} and t_o is the time required to reach the threshold damage state. The per cent swelling due to the production of voids is then,

$$\frac{\Delta V}{V}(\%) = (Kt)SF \qquad (1)$$

where

S is a function of the material properties such as sink density and size includes the dislocation density

and

F is mainly a function of the irradiation temperature, the migration energy of the vacancies and has a weak, complex dependence on K.

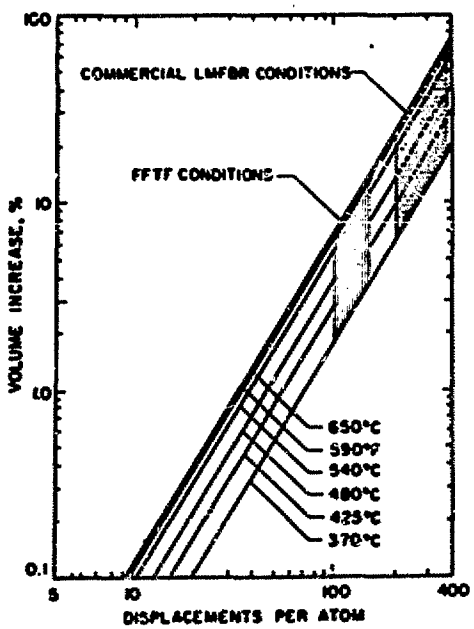


Figure 1. Swelling Estimates of 20% Cold Worked 316 Stainless Steel FFTF Interim-Design Basis (August 1969) Reference 8.

Although this theory was only fully developed in the last few years, Nelson and Mazey (1) recognized one of its key features in 1969. It is noted that once nucleation has been accomplished, the swelling is proportional to the product of K and t and does not, to a first approximation, depend on these two quantities separately. This means that if one wants to shorten the irradiation time, then it is only necessary to raise K. The early experiments used high intensity beams of heavy ions whose displacement effectiveness per particle is approximately 10^2-10^6 times that of equivalent neutron fluxes (Figure 2). When this displacement effectiveness was multiplied by currently available beams of charged particles, scientists were able to raise the displacement rates by factors of approximately 10^3-10^4. By using such high displacement rates, the irradiation time required to reach a given damage state was lowered by the same amount and designers could then have reasonable swelling information in a matter of days compared to the many years required for neutron irradiation.

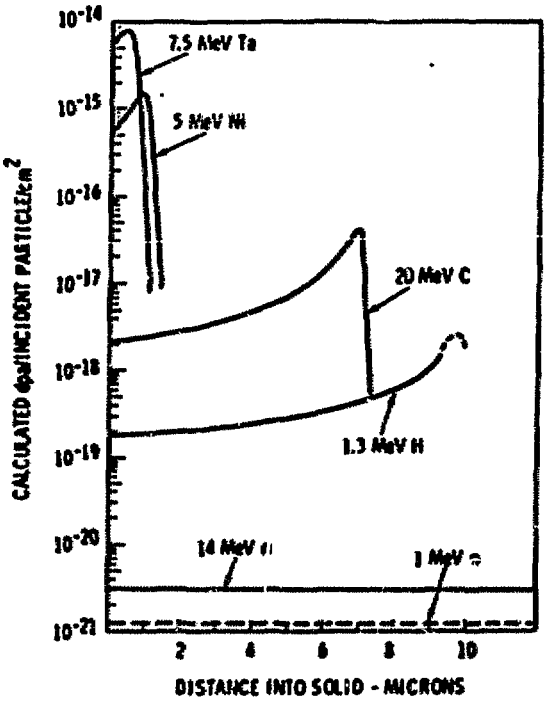


Figure 2. Displacement Effectiveness for Various Energetic Particles in Nickel.

There is one correction that must be made because of the high displacement rates and that is the effect of K on F in equation 1. It was recognized by Bullough and Perrin (10) that the important quantity that must stay constant in ion bombardment experiments is

$$\frac{K_1}{D_{v1}} = \frac{K_2}{D_{v2}} \qquad (2)$$

where

when K_1, K_2 are the defect production rates in two different irradiation environments.

D_{v1}, D_{v2} are the diffusivities of vacancies during the experiments and proportional to exp $-(Q_m^v/kT_{1,2})$

This means that slight adjustments in the irradiation temperature would have to be made to maintain the equality in equation 2. The calculated peak temperature shift, while perhaps too simplified in terms of more recent theories but nevertheless still useful, is shown in equation 3.

$$\frac{T_1 - T_2}{T_2} = \frac{\left[\frac{kT_2}{Q_m^v} \ln\left(\frac{K_1}{K_2}\right)\right]}{\left[1 - \frac{kT_2}{Q_m^v} \ln\left(\frac{K_1}{K_2}\right)\right]} \qquad (3)$$

The significance of equation 3 is that in order to simulate damage at some displacement rate K_2 and temperature T_2 by irradiation at a higher displacement rate of K_1, one must irradiate at a slightly higher temperature T_1. We will see an example of this shift later in the paper but Figure 3 from Brailsford and Bullough (11) shows the effect of K on F for a factor of 10^3 difference in displacement rate.

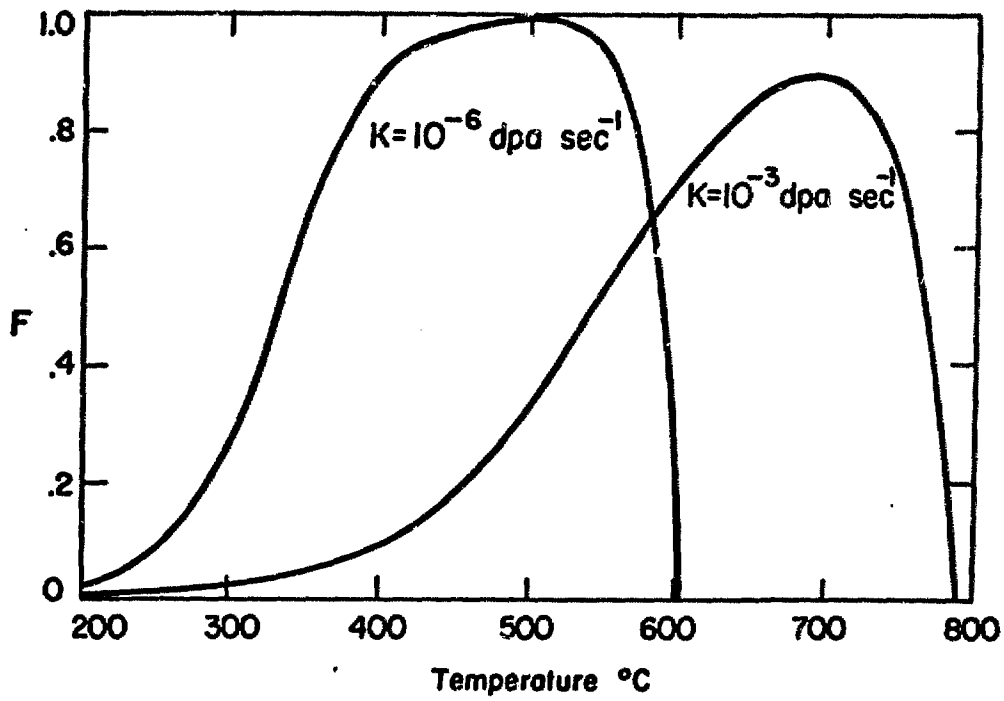


Figure 3. Effect of Displacement Rate on the Temperature Dependence of Void Swelling in Metals-Brailsford and Bullough (11)

Types of Simulation Studies Which Have Been Performed

An attempt has been made to summarize in Table 1 all of the simulation studies reported to date. Chronologically, the earliest studies were performed with relatively low energy (~100 keV) beams of H, C, O, and Fe at Harwell (1). These were quickly replaced with 20 MeV carbon ion studies (23-29) which were carried on for 1-2 years before switching to a 45-50 MeV Ni ion beam which is used almost exclusively now. Investigators at Atomics International started soon after the initial Harwell studies with beams of 0.75 to 1.4 MeV protons (30-35). Their work has been almost entirely directed toward the austenitic stainless steels. Scientists at Battelle Northwest Laboratories were the first to use high energy (5-11 MeV) heavy ion beams of Cu, and Ni on the austenitic stainless steels (35-39) but their work is now directed toward pure metals,

Table I
Summary of Continuing Ion Bombardment Studies
Directed at Void Formation

Laboratory	Ions	Ion Energy-MeV	Materials Studied	Reference
Harwell	H,C,O,Fe	0.1	316SS	1
	C	0.1	Co	14
	C,Cu,Ni	0.1	Ni,Cu,Ni-Cu	15
	Al	0.4	Al	16
	N	2	Mo,TZM	17-22
	C	20	316SS,Ni,PE16	23-29
	Ni	46	Ni,Ni alloys	29
AI	H	0.75-1.4	304SS,316SS,321SS	30-34
	"	"	Ni	35
	"	"	Ta	34
BNW	Cu	5	316SS	35-37
	Ni	5	316SS	38,39
	Se	6-11	Ni	40-42
	Ni	5-6	Ni,Nb,Mo,TZM	39,42-46
	Ta	7.5	Mo,Nb,V,TZM, Nb-1Zr,Ta	42-48
Saclay	Ni	0.1-0.5	Ni	49-51
	Cu	0.5	Cu	49,52
ANL	H	1.25	304SS	53
	Ni	3.5-4	304SS,316SS,321SS	53-55
	Ni	3.25-4	V,V alloys	56-57
	Ni	3	Nb	58
G. E.	D	12.3	304SS,321SS	59
	Ni	5	304SS,316SS,321SS	60-65
NRL	Ni	2.8	Ni,Fe,Fe alloys	66-67
Wisconsin	Al	2	Al	68
	Cu,Nb	15	Al,V	69

i.e. Ni, Nb, and Mo (39-46). A 7.5 MeV beam of Ta ions was also used to study refractory metals and alloys and currently the Ni and Ta beams are used almost exclusively (43-48). A group at Saclay, headed by Adda, reported using 0.1-0.5 MeV beams of Ni and 0.5 MeV beams of Cu on Ni and Cu respectively and this work is still continuing (49-52). Another group at ANL has reported the use of 1.25 MeV hydrogen (53) and 3.5-4 MeV beams of Ni on austenitic stainless steels (53-55). More recently the 3-4 MeV beams of Ni ions has been used to study void production in V alloys (56,57) and Nb (58). Initial work at G. E. utilized a 12.3 MeV beam of deuterium ions to bombard austenitic stainless steels (59) but a large amount of work has recently been done with a 5 MeV beam of Ni ions from the same accelerator that the BNW group used at High Voltage Engineering Corporation. (60-65) More recently a group at Naval Research Laboratory has used a 2.8 MeV beam of nickel to study Ni, Fe and Fe alloys (66,67). Work at the University of Wisconsin has been started using a 2 MeV beam of Al (68) and a 15 MeV beam of Cu and Nb ions to study pure Al and V (69).

Irradiation, Analysis and Calculation Techniques

The use of high energy charged particles from accelerators to produce high damage states in materials has been treated previously (23,30,39). One drawback (or advantage depending on the type of study) to ion simulation work is the necessity to supply gases to the solid which are normally generated neutronically. Preinjection with helium to 1-100 ppm has been described by several authors and the reader is referred to these references (59,70,71). While it is common practice to preinject the samples with helium before irradiation, such a practice must be closely studied, especially at high helium contents because it may influence the nucleation at an early stage of irradiation. A much better technique would be to inject simultaneously with helium from an auxilliary accelerator.

Because of the limited range of the charged particles, the analysis of the microstructure by transmission electron microscopy (TEM) is often a time consuming task. Methods for precise removal of damaged layers and subsequent analysis of the size and density of voids have also been described in the literature (23,30,39,41, 72). An interesting technique which can measure overally swelling without resorting to precise TEM analysis has been described by Johnston et.al., (60-62). Their technique relies on measuring the difference in the step height of a surface which has been irradiated as opposed to a surface which was shielded during irradiation. The bombarded area tends to swell and protrude from the surface in relation to the shielded region (Figure 4).

Finally, the correlation between neutron irradiation work and ion bombardment studies is made through the common unit of damage

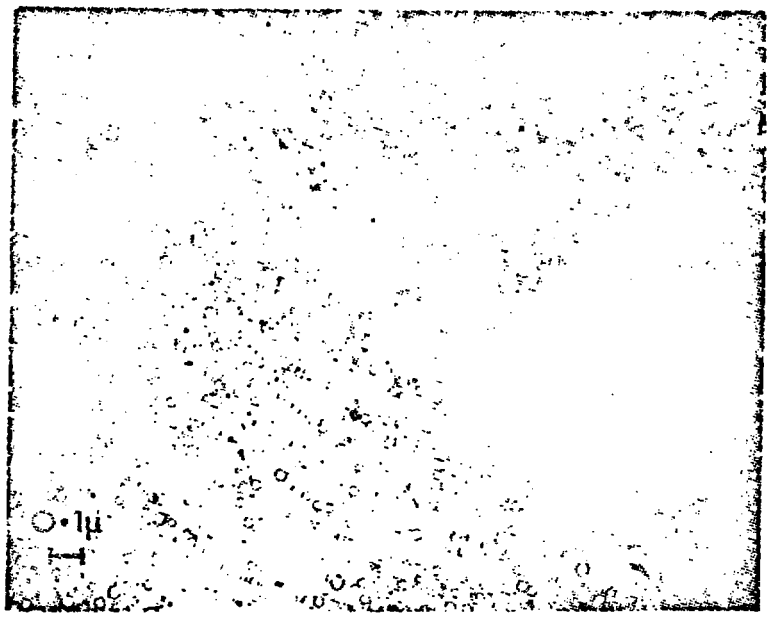
Figure 4. Surface of a 304SS Specimen that was Covered with a Mask Containing Diamond Shaped Holes During 5 MeV nickel bombardment at 660°C. The bombarded regions have been elevated as a result of the swelling (Reference 61).

called the dpa, for displacements per atom. Methods for calculating dpa values differ from group to group and standardization in this field is sorely needed. Nevertheless the reader can obtain some idea of how the damage is calculated in various ion bombarded samples by reading references 19,23,54,62,73-75.

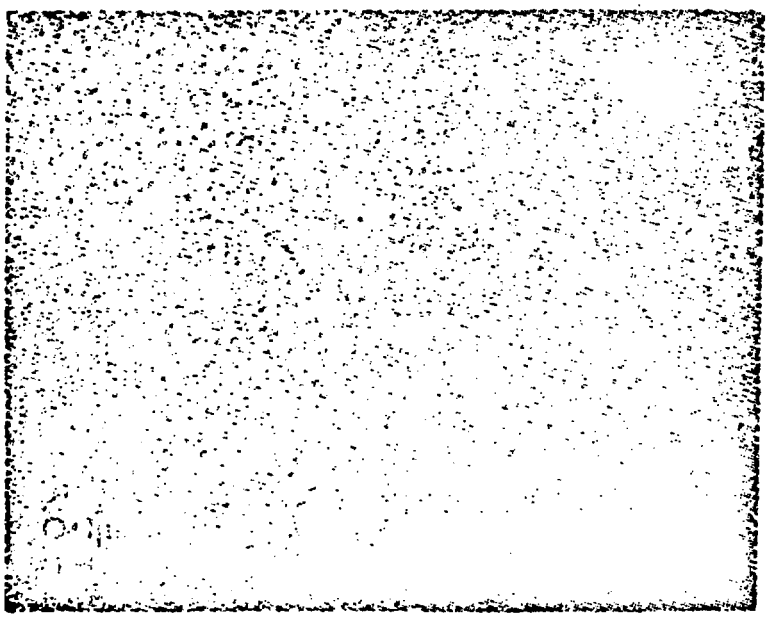
Some of the Noteable Successes of the Ion Bombardment Technique

One of the earliest conclusions of the simulation studies had to do with the role of gases in nucleating voids in metals. It was known that helium atoms were generated in reactor components and it was normally assumed, prior to 1969, that these gas atoms were the nuclei for producing voids in metals. The simulation studies provided the first unambiguous test of this theory because the experimenter could choose to dope with or exclude helium from the sample during the experiment. Early studies revealed that helium gas was not required for void nucleation in steels, Ni, Mo, V, and Nb (1,17,23,36,37,43). On the other hand if helium was present, it could have a very dramatic effect on the nucleation as shown in Figure 5. More recent studies by Johnson et al. (60,61,63-65) have shown that in stainless steels, samples containing helium gas swelled more than those that did not. In a somewhat unrelated study, Loomis et al. (58) found that in the absence of helium, void nucleation was inhibited by increasing amounts of oxygen impurities in Nb.

Another early success of this technique was the discovery of the void superlattice in several pure materials; Al (16), Ni (41,42,46), Nb (42,44-47), Mo (17-22, 46,48). It was found that if pure metals are irradiated to a high damage state (>>1 dpa)



0.1 ppm He



100 ppm He

Figure 5. Effect of helium on void nucleation in 316 SS at 550°C during 20 MeV carbon ion bombardment. Damage level is 15 dpa (23).

near the temperature threshold for void formation (~0.3T_m) that the voids would form arrays consistent with the crystal structure of the parent lattice. For example, voids in N^+, Ni^{++} or Ta^{++} bombarded Nb and Mo formed a bcc array (Figures 6 and 7) and those in Al and Ni formed fcc superlattices. This unique structure was discovered at about the same time an equivalent structure was found in neutron irradiated metals by Wiffen (76) and Erye (77). Since none of the ion bombarded samples contained preinjected helium it was clear that helium was not responsible for the observed effect. It is also evident that the ordered void structure is not peculiar to high damage rate processes because those formed during neutron irradiation did so at a damage rate of 1/1000 to 1/10,000 that of

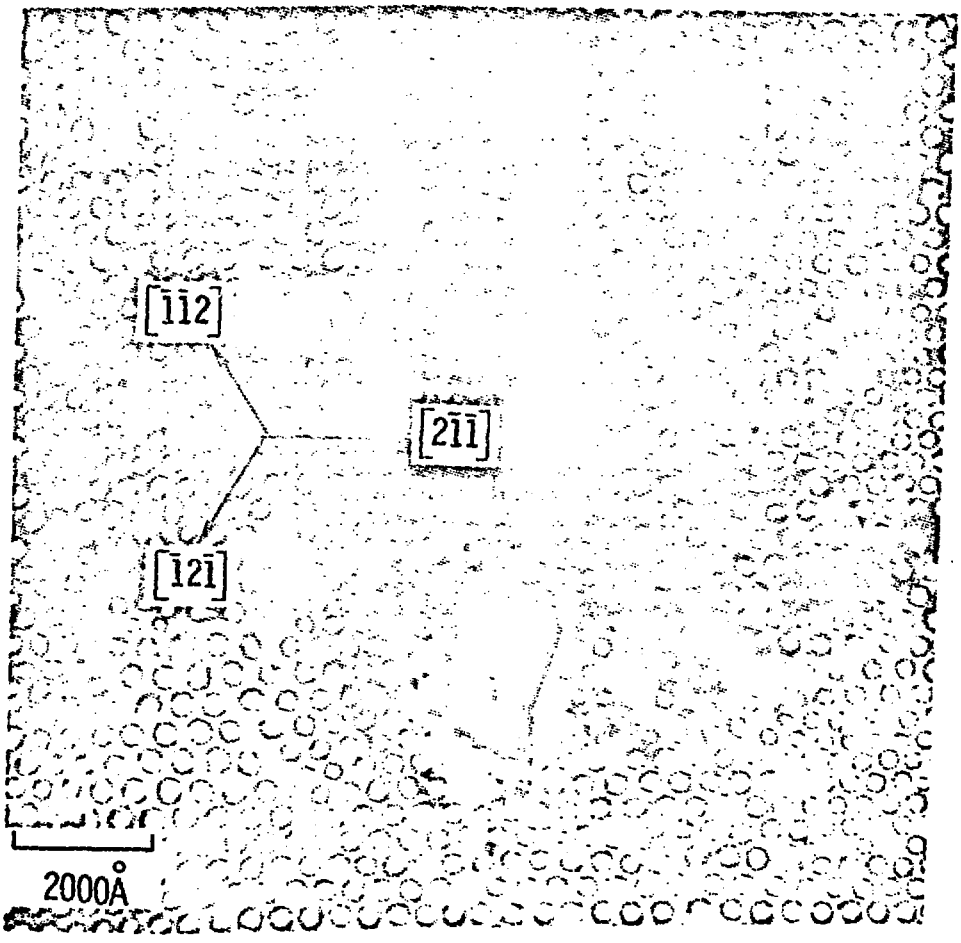


Figure 6. Superlattice of voids in Nb bombarded at 800°C to 300 dpa with 7.5 MeV Ta ions. The superlattice is being viewed along the <111> direction and is bcc in nature (46).

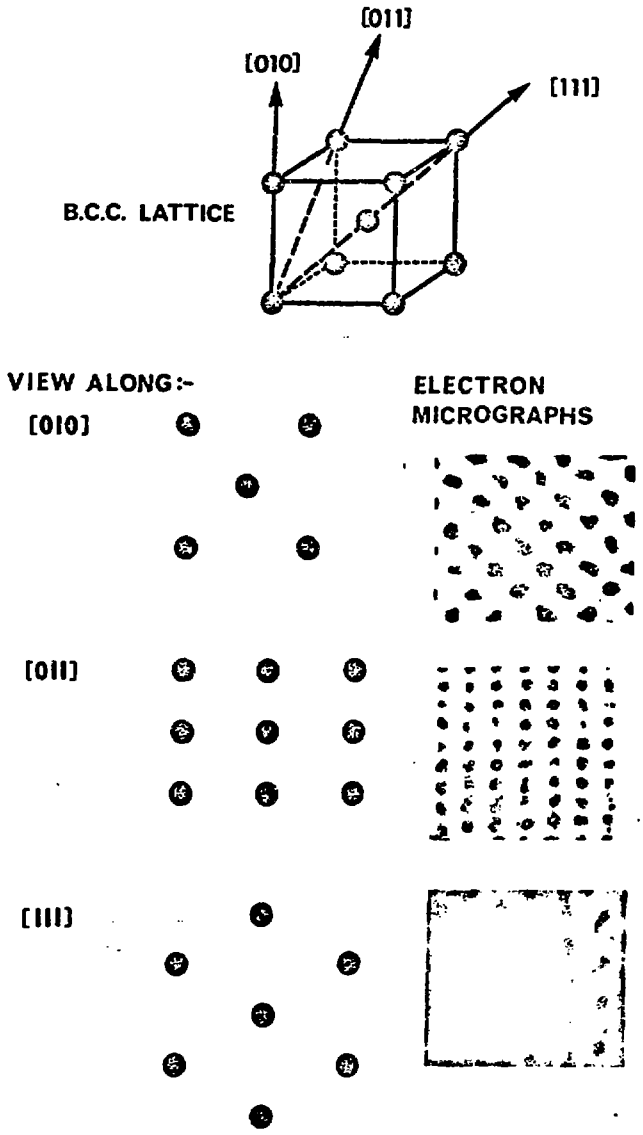


Figure 7. Superlattice of voids in Mo bombarded at 870°C to 100 dpa with 2 MeV nitrogen ions. The various projections of the bcc superlattice are shown (20).

the ion bombardment studies. There are several current theories to explain the superlattice formation (78-81) but the exact situation is not clear at this time. Updated information from a recent review article (46) on ordered structures is given in Table II.

Another area which ion bombardment studies have proved to be extremely important is that of swelling saturation. Because of the extremely high damage rate in ion bombardment experiments it was quite easy to surpass the damage levels of the present and get a preview of what might happen at extremely high damage states. Early work by Harwell and BNW (25,41,42) showed a saturation in the swelling behavior of Ni (Figure 8) and the Harwell work even showed a saturation in swelling behavior of 316 stainless steel (25) (Figure 9). Later work has shown a similar saturation in Nb(47) (Figure 10).

The cause of the saturation is not clear at this time, but the onset of saturation corresponds to the formation of a superlattice in the case of pure Ni and Nb. It is speculated that when the density of voids reaches such a high level, the dislocations are absorbed into the voids thus removing the bias for void formation. The close spacing of the voids also makes then the predominant sinks for both interstitials and vacancies thus prohibiting void growth.

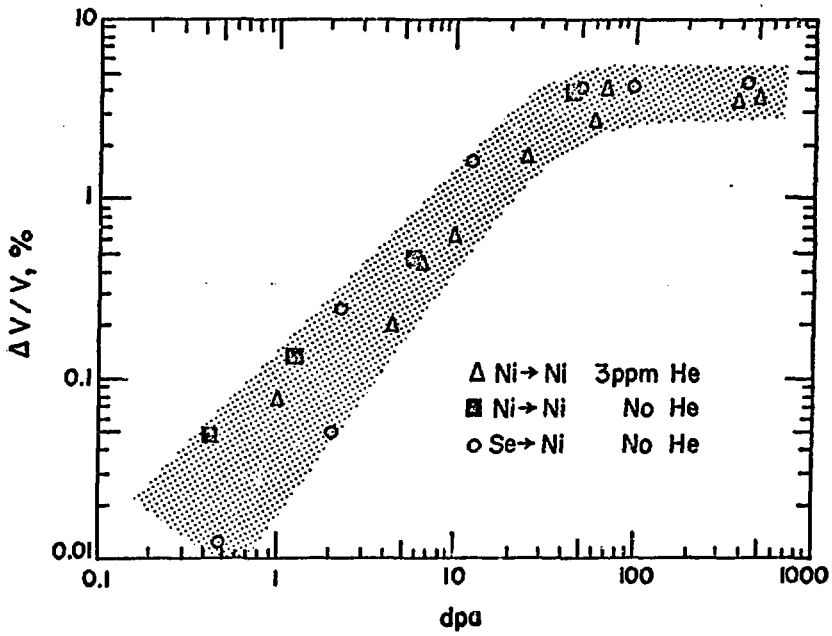


Figure 8. Saturation in swelling of Ni after heavy ion bombardment (42).

TABLE II

SUMMARY OF DATA ON ORDERED VOID LATTICES

Material	Bombarding Species	Irradiation Temp. °C	Damage Level dpa	Super-lattice Structure	a_o Void Lattice Constant Å	$2r_v$ Ave. Void Size Å	$\frac{a_o}{r_v}$	Reference
Ni	5 MeV Ni	525	360	fcc	620	250	5.0	41,42
Ni	6 MeV Se	525	400	fcc	660	180	7.3	41
Al	400 KeV Al	50-75	40-80	fcc	600-800	100-140	8.5-12	16
Mo	neutrons	585	36	bcc	265	64	8.3	76
Mo	neutrons	790	36	bcc	328	72	9.1	76
Mo	neutrons	650	30-60	bcc	340	40	17	77
Mo	2 MeV N	870	100	bcc	220	40	11	20
Mo	7.5 MeV Ta	900	130	bcc	310	60	10	46
Mo (a)	7.5 MeV Ta	900	150	bcc	460	140	6.6	48
Mo-0.5 Ti	neutrons	585	36	bcc	215	69	6.1	76
Mo-0.5 Ti	neutrons	790	36	bcc	315	72	8.8	76
TZM	2 MeV N	870	400	bcc	220	60	7.3	20
Nb	7.5 MeV Ta	800	140	bcc	340	125	5.4	46
Nb	7.5 MeV Ta	800	290	bcc	380	110	6.9	46
Nb	7.5 MeV Ta	900	300	bcc	750	250	6.0	46
Nb	5 MeV Ni	800	5	bcc	350	45	15.6	42
Nb	neutrons	790	34	bcc	665	186	7.2	76
Ta	neutrons	585	20	bcc	205	61	6.7	76
W	neutrons	550	15	bcc	195	30	13	84
W	neutrons	800	15	bcc	250	40	12.5	84

a) single crystal

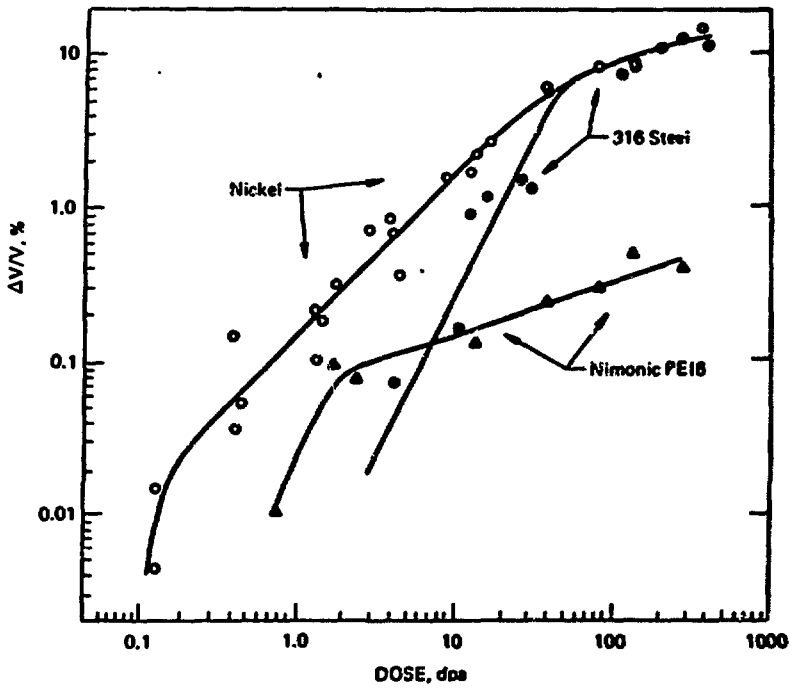


Figure 9. Swelling behavior of Ni, 316 SS and PE-16 during 525°C bombardment with 20 MeV carbon atoms. All materials contain 10 ppm He. (25)

This reasoning does not hold true for the saturation in 316 stainless steel because no void superlattice has been found in this material. There has been a recent suggestion that the carbon atoms which are producing the damage may build up to such a level as to suppress the void formation. Recent work by Steigler et al. (82) on very low carbon containing 316 stainless steel would tend to support the carbon suppression theory. The discrepancy could be solved by irradiating to very high fluences with self ions. Such experiments have been performed by Johnson et. al., (63-65) and the ANL group (53-55). Neither of these latter groups finds such a saturation (Figure 11). The lack of a saturation in steel is also supported by proton work at AI (34). However, the possibility exists that in the proton experiments hydrogen may be promoting swelling before it diffuses from the sample. Finally, recent studies of 316 stainless steel by high voltage electron microscopy (83) (HVEM) also show a continuing swelling with fluence. Considerable work is needed here before one will be able to confirm or deny the saturation phenomena in steel.

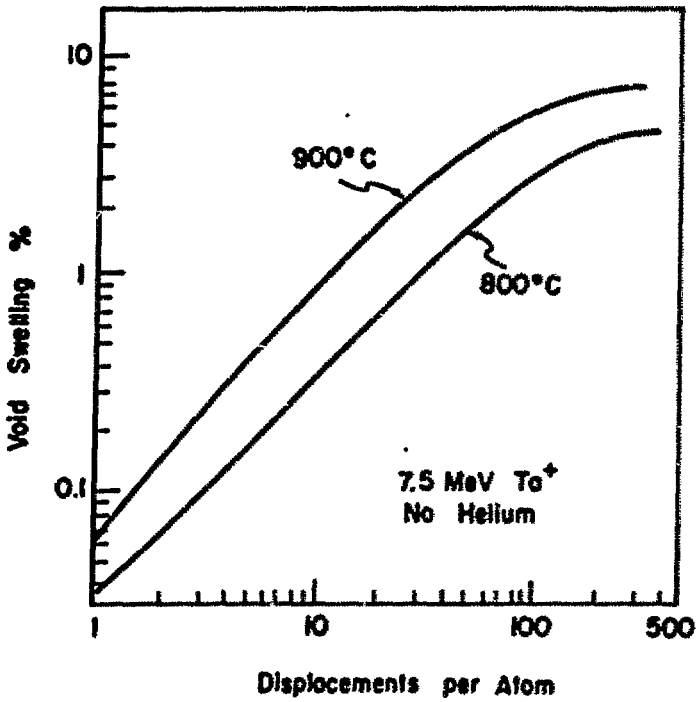


Figure 10. Saturation of swelling in Nb during 7.5 MeV Ta ion bombardment (47).

Probably the most important success of the simulation technique from an economic standpoint is the extension of present swelling information on fast reactor cladding. The four most active groups in this area are Harwell, G. E., AI and ANL and some of their results are shown in Figures 9,11-12. Harwell and G. E. have also used the simulation technique to screen various alloys with respect to their swelling behavior and some typical results are shown in Figure 12. The effects of thermomechanical treatments such as cold working 316 SS (64) (Figure 12) and formation of coherent precipitates in PE16(25) (Figure 9) have been shown to be quite beneficial in reducing the swelling behavior. The fact that one can study several alloys within the period of a few days at the same closely controlled conditions, means that large, costly, and time consuming screening programs with radioactive metals can be avoided.

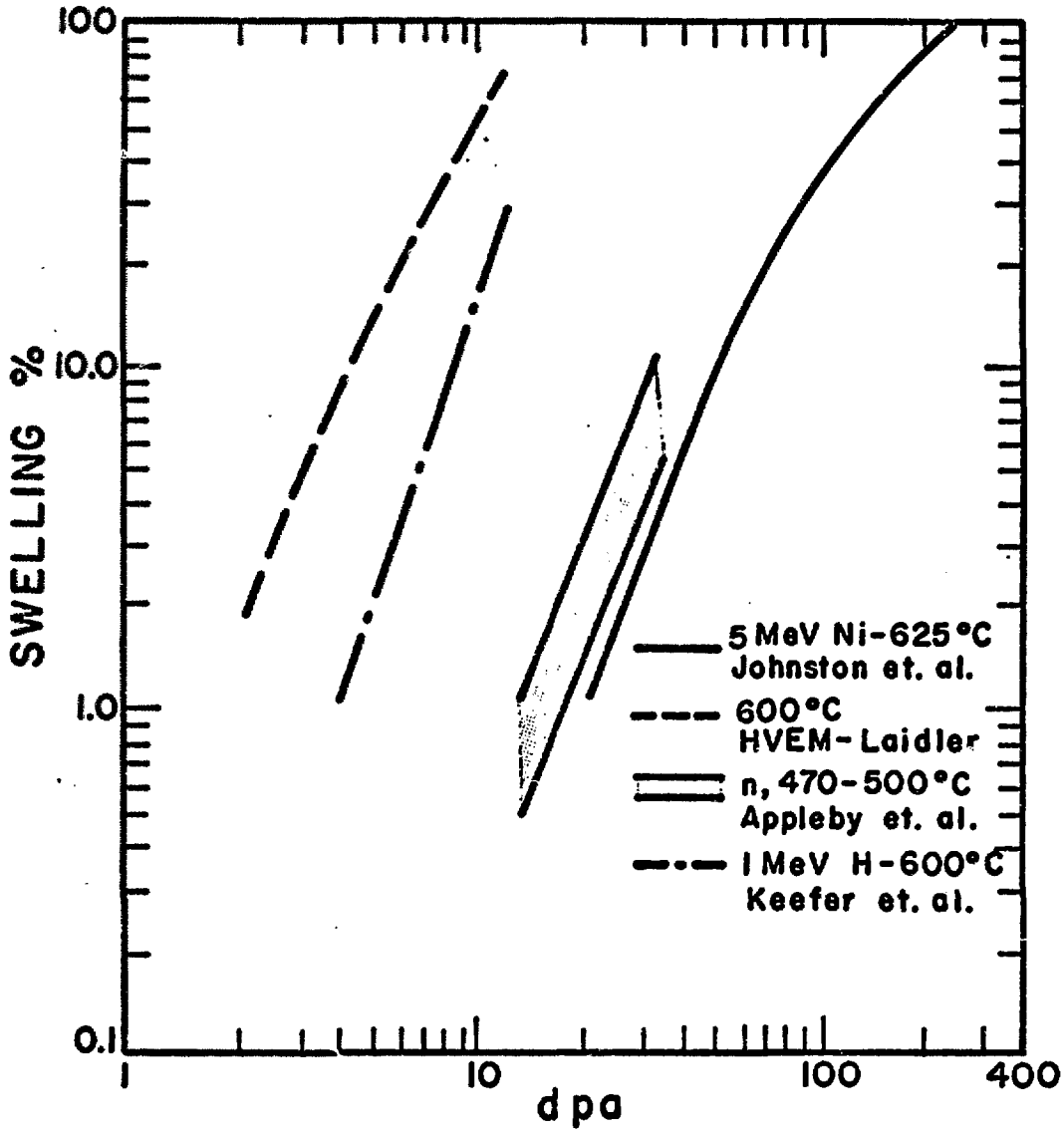
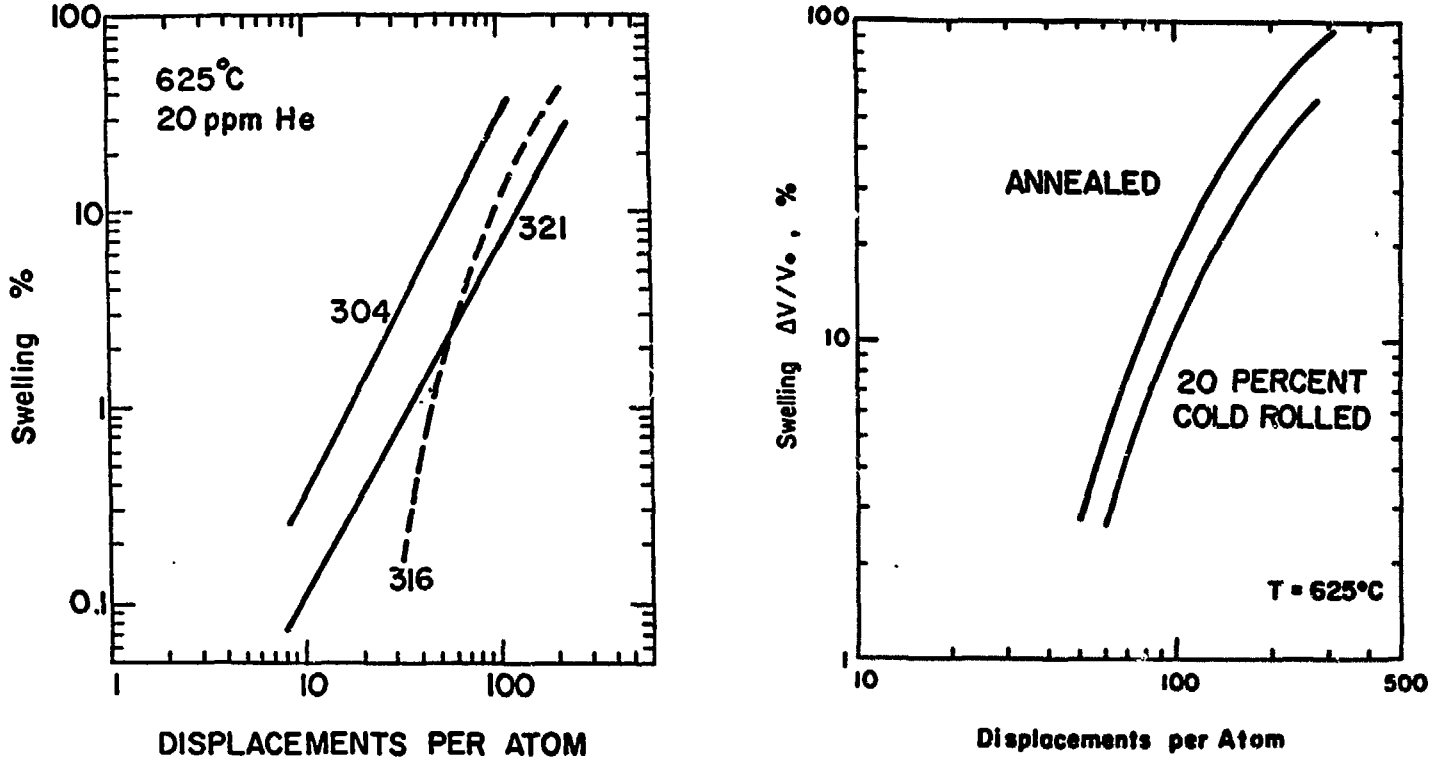


Figure 11. Comparison of actual neutron induced swelling in ST 316 SS to that determined by H+, Ni+, and HVEM simulation studies. Note that the ion bombardment studies were conducted at a high enough temperature to overcome the temperature shift. The dpa values were corrected to recent recommendations by D. G. Doran for protons, electrons, and neutrons (47 dpa per 10^{23} n/cm^2, E > 0.1 MeV).



(a) (b)

Figure 12. Example of alloy screening by 5 MeV Ni ion bombardment (a) Intercomparison of alloys (61); (b) Examination of cold work (64)

Once promising alloys are identified, concentrated study under reactor conditions can be pursued. Herein lies one of the greatest promises of this simulation technique.

The group at Harwell has used this approach in coming up with a more swelling resistant alloy, PE16, a nimonic nickel based alloy. It can be seen from Figure 9 that this alloy swells almost a factor of 30 less than solution treated 316 stainless steel at a 525°C irradiation temperature.

There have been other discoveries in this field which deserve mention here and will undoubtedly be investigated more fully in the future. These include the early detection of a reversion of the austenitic phase to ferrite at high damage states which could have severe implications if the trend continues (31). Several groups (BNW, ANL, NRL, and Harwell) are investigating the effects of alloying on void suppression for non LMFBR materials. These studies could lead to a better fundamental understanding of the void formation process in all metals. Finally, a group at NRL has discovered that rastering the ion beam across the surface of a sample can give results completely atypical of defocussed beam conditions (66). The effect can be traced back to the fact that even though the average dpa rate is the same for both samples, it can be considerably higher instantaneously and locally in the rastered sample. Void formations under extremely high dpa rates can have technical significance in future fusion reactors as we shall see later.

Limitations of the Ion Bombardment Technique

The major limitations that are known at this time fall into 5 categories:

1. Alloying
2. Stress
3. Temperature shift
4. Lack of Mechanical Property Data
5. Excess Interstitials

We have already discussed some of the possible problems with respect to a change in chemical composition. The experimenter must constantly guard against this problem especially if he wants to make any sense out of temperature or fluence dependence of the void size or density. Normally, TEM analysis is performed at a position far from the end of path of the incident ion in order to minimize the possible contamination effects. One must be especially careful about studies which use ions which are mobile during irradiation because they could migrate from the point of deposition to areas of TEM analysis.

Another complicating feature of the ion studies is that because of the limited range of the atoms (on the order of a few microns at most) the majority of the sample is undamaged. When considerable swelling is induced, the undamaged part of the sample can impose large constraints on the expanding region. Such effects are quite evident in Figure 4 and the effect of stress on swelling must be understood before the magnitude of this problem can be assessed in ion bombarded samples.

We have already seen that one must raise the ion bombardment temperature with respect to that being simulated because of the rate effect. The higher temperature irradiation could give atypical results if this temperature causes microstructural changes in the material not seen at the temperature to be simulated. A good example of this effect can be seen in 20% cold worked 316 stainless steel. Straalsund and Brager (85) have shown that 100 hours at 650°C is sufficient to cause some recovery of the cold work in 316 stainless steel. Since the high dislocation content tends to suppress void formation, its removal during high temperature ion bombardment studies may give misleading results. A typical temperature shift for a displacement rate ratio of 10^3 in steel is approximately 100-150°C which means that IB studies would probably be limited to simulating temperatures less than 500-550°C during neutron irradiations. Similar reservations must be stated for alloys with complex precipitates that could be dissolved at high temperature.

The limited range of charged particles also means that the samples cannot be used for mechanical property measurements. Even the 45-50 MeV Ni ions used by the Harwell group have only a 5 micron range in steel (29). Perhaps the only chance for such measurements will be the high energy (>20 MeV) proton or deuteron beams such as those used at ANL (86).

One final concern that must be mentioned for self ion irradiation (i.e. Ni→Ni or SS) is the addition of excess interstitials to those already produced by irradiation. It has been speculated that if the ratio of injected atoms to displaced atoms is more than 1:100, the biasing effect of the dislocations could be overcome and the swelling suppressed. However, such an effect has not been seen in studies conducted to date.

Possible Directions For Future Simulation Studies

It is expected that the majority of simulation studies in the immediate future will be aimed at predicting fast reactor cladding behavior. Particular attention will be paid to the potential saturation effects and the screening of alloys for advanced reactor concepts.

Basic studies will probably concentrate on the effect of metallic impurities and gas atoms on the nucleation of voids in metals. These studies should be able to contribute significantly to the development of a complete theory of void swelling in metals.

The study of void superlattices should lead to a consistent theory for their formation. It is hoped that such theories may also be able to predict uses for ordered structures in such areas as superconductivity (i.e. flux pinning sites), high strength metals or for studying the interaction of dislocations with a regular array of defects. One interesting feature of this work is that such ordered arrays can be formed quickly, are quite stable thermally, and could be formed in metals without introducing radioactivity.

The advent of fusion reactor technology will certainly benefit from this simulation technique. Alloy screening studies will help narrow the potentially promising alloys to a few which can then be studied in depth when 14 MeV neutron test facilities become available. The generation of gas in such a hard neutron spectrum is truely a unique problem for fusion reactors as seen in Figure 14 where the ratio of the helium gas to dpa values are given for V in thermal and fast fission reactors as well as for fusion facilities (87). It can be seen that gas to dpa ratio in fusion reactors is 2 to 3 orders of magnitude higher than in fission reactors which means that it will not be possible to simply test potential fusion reactor materials in fission facilities. However, by coupling two accelerators, one generating helium ions and the other generating heavy ions it will be easy to generate the proper ratios typical of any nuclear facility.

Finally, and perhaps one of the most promising future areas of study will be the simulation of extremely high damage rates typical of pulsed or laser fusion reactors (Figure 15). Steady state magnetic reactors will have damage rates comparable to current fission facilities ($\sim 10^{-6}$ dpa sec^{-1}) (87) but pulsed systems will have instantaneous rates of $\sim 10^{-5}$ to 10^{-4}/dpa sec^{-1} separated by 3 seconds of no damage at all (88). This is even more aggravated in laser systems where the damage rates are ~0.1 dpa sec^{-1} for approximately 1 microsecond. Such laser systems may be pulsed on the order of 10 times a second (89). It will be very risky to extrapolate the current radiation damage data over such a wide range of damage rates and much theoretical and experimental work is needed to assess this situation.

Conclusion

It has been shown that in the short period of four years the ion bombardment simulation technique has made notable discoveries

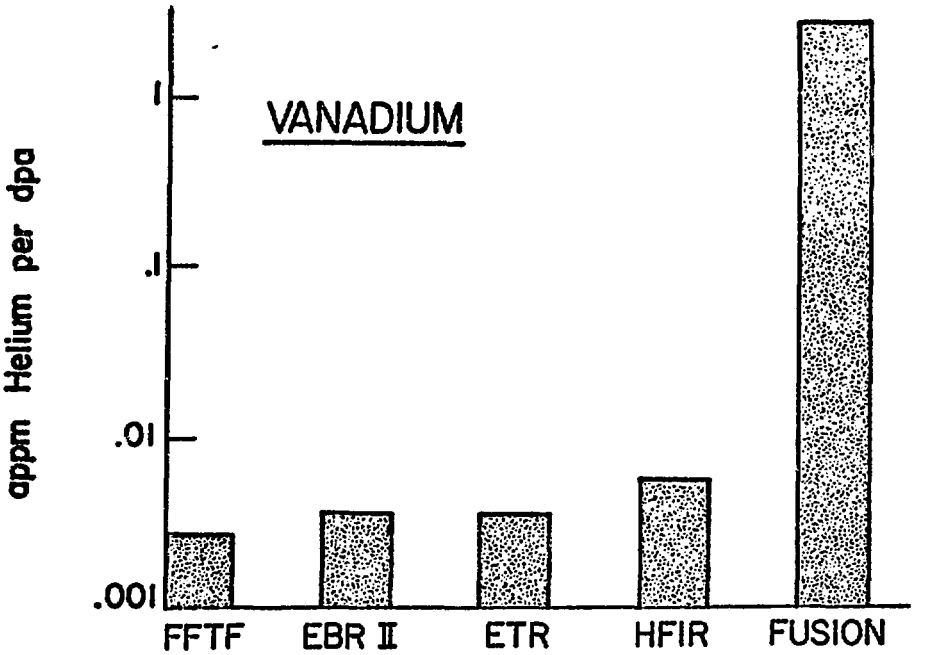


Figure 13. Ratio of the atomic parts per million of helium gas generated per dpa in vanadium for various nuclear facilities (80).

about the formation of voids in metals. The simulation studies are now giving us a preview of what to expect after long term high temperature neutron bombardment and it is a quick, easy, and economical tool to screen new alloys. Future work of a basic nature will undoubtedly contribute to void nucleation and growth theories. It is possible that the simulation technique will be even more valuable to the controlled thermonuclear reactor program than it has been to the LMFBR program.

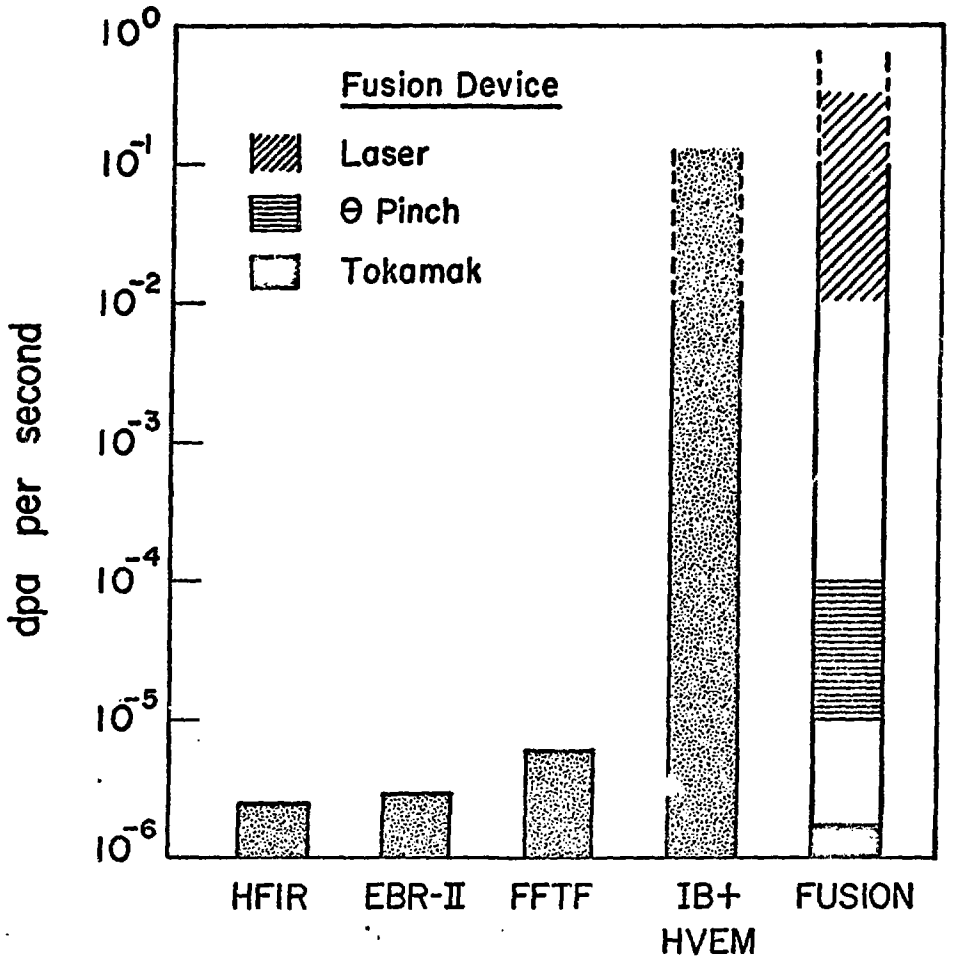


Figure 14. Representative displacement rates in 316 SS for several nuclear facilities. Note that instantaneous dpa rates for fusion reactors can be quite large even though a time average value is 10^{-6} (87).

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