AN APPLICATION OF CYCLOTRON TO RADIATION DAMAGE STUDY FOR FUSION REACTOR MATERIAL DEVELOPMENT

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SUMMARY

Necessary conditions for fusion neutron radiation damage simulation with ion beam bombardment is outlined. Brief literature survey on works in this field are given. The NRIM cyclotron facility which started irradiation creep experiment this May is described. Some preliminary results on irradiation creep and helium embrittlement are shown.

Introduction

Fusion reactor is now being developed in the world major countries. Critical condition of D-T plasma for self ignition will be realized within a few years and development of reactor technology must be emphasized in the next step. D-T reaction produces 14 MeV neutron. This high energy neutron causes severe radiation damage in material. For the reactor core design, the radiation damage must be appropriately evaluated and the acquisition of extensive data base is necessary. The recent works on radiation damage has shown that present commercial materials such as austenitic stainless steels and nickel base heat resisting alloys can not be used in such severe neutron environment as in the commercial fusion reactor and new radiation resisting materials are highly desired.

Requirements for simulation

<u>Displacement damage</u> As illustrated in Fig. 1, high energy neutron produces two types of radiation damage in material. In one type, a lattice atom is displaced from its lattice site. When this primary knock-on atom has high kinetic energy, it can produce a cascade damage. when the transferred energy to the primary knock-on atom is much lower, it only produces more simple defects of lattice vacancy and interstitial atom known as Frenkel pair.

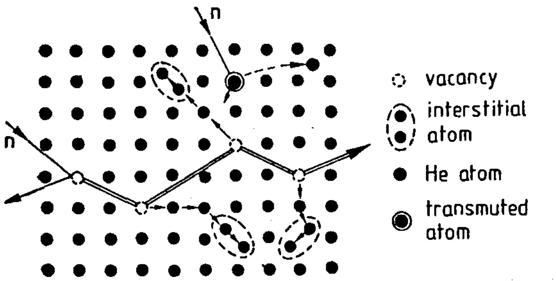

Fig.1 Schematic illustration of interaction of high energy particle with a metal lattice atom.

<u>Transmutation</u> Another type of radiation damage is transmutation by nuclear reaction. In view of material property degradation, helium production by (n,alpha) reaction has particular importance. For example, it is anticipated that more than 1,000 at.ppm helium can be produced in 316 stainless steel in the commercial fusion neutron environment.(1)

<u>Secondary radiation damage effects</u> The displacement damage causes hardening and embrittlement, void swelling and irradiation creep in structural material, for example, in first wall material. The produced helium results in also bubble swelling and embrittlement, especially at high temperature.

<u>Energy spectrum of knock-on atom</u> The cross section which produces high energy part of the primary knock-on atom and thus cascade damage is similar between 14MeV neutron and light ion.(2,3) Disadvantage of the light ion is that because of Rutherford scattering, there are formed fairly larger number of single defects.(4,5) It is considered that this production ratio of cascade to single damage is essential to develop defect structures such as dislocation loop and void. This point must be cleared out theoretically.

<u>Higher helium production</u> Except for several element of Ni,Cu,B, one to two order higher concentration of helium is generated in fusion reactor neutron environment, than in fission reactors.(1)

<u>He/dpa ratio</u> The ratio of at.ppm He produced with (n, alpha) reaction to displacement damage in dpa unit is used to characterize neutron environment. In fast breeder reactor, this value is order of 0.1, but 10 to 20 in fusion reactor for austenitic stainless steel. So, it is not possible to estimate material property change in the fusion environment with the fission reactor, except for some special cases such as stainless steels irradiated in HFIR including nickel as alloying element. The effect of this critical at.ppm He/dpa value can be estimated in light ion irradiation, though only at low fluence.

<u>Simulation with light ion</u> In any simulation technique, these two factors of displacement damage and transmutation must be evaluated correctly. The fundamental aspects of simulation with light ions are schematically shown in Fig.2.

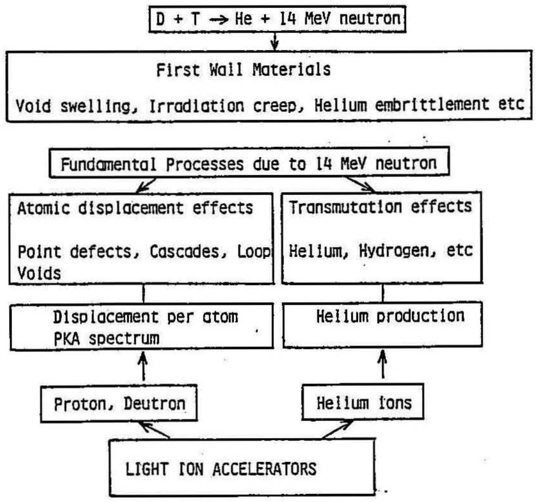

Fig.2 A cyclotron application to the study of radiation damage for fusion materials development

Proton and deuteron beam bombardment with energy of 10 to 20 MeV can simulate the cascade damage well(2,3),but Rutherford scattering in them produces a lower tail in the primary knock-on atom energy spectrum and thus, more Frenkel type defects,as mentioned above. This results in higher concentration of free vacancy and interstitial which diffuse in long range and produce somewhat different secondary defect structures. This difference must be taken into consideration when one correlates neutron and light ion damage experiment. Helium effect can be simulated with (p,alpha) reaction and helium implantation. So, light ion bombardment can be a useful method for estimation of material behaviour under fusion neutron irradiation,though the low energy spectrum problem and difficulty to obtain high fluence irradiation limit the applicability. Other simulation methods such as 14 MeV neutron source, fission reactor, heavy ion irradiation and electron irradiation with transmission electron microscope have also merit and demerit respectively. At present, it is considered to be best way to use all these methods complementarily and in combination.

Survey of literatures

Advantage of light ion application, compared with heavy ion, is a possibility of mechanical properties evaluation. The applications of light ion are divided into two categories. One is irradiation creep and the other is helium implantation. Numbers of review papers were written already(6-17). This paper mainly covers the areas on works being intended at NRIM now.

Irradiation creep This type of experiment was started in early stage of 1970's, first at ANL and about ten research institutes have followed it. Now, several institutes in Europe and Japan continue, but several institutes in USA and AERE Harwell interrupted the experiment. The effects of experimental conditions such as fluence, flux, pulse beam, stress, and temperature have been investigated. Commercial alloys such as 316 SS and Zircaloy, and experimental nickel and its alloys were main concerns. Comparison between neutron and light ion irradiation is an interesting point in view of creep mechanism and validity of simulation. Some results are summarized in Table 1. In this table, results on similar type experiment using Van de Graaf are included.

Helium implantation The mechanism of helium bubble growth and the effect of applied stress on it were investigated with helium preimplantation and anneal method. Precipitation of helium bubble, especially on grain boundary, causes severe embrittlement. This phenomenon was initially investigated with tensile test after helium implantation. Now creep rupture test during helium implantation is carried out. This type of experiments are summarized in Table 2.

Application to other type tests In light ion irradiation, it is possible to obtain bulk specimen. Extensive studies on property changes such as electric resistance, internal friction,Mossbauer spectrum, positron annihilation and so on are carried out.These results are also included in Table 1 and 2.

Higher energy proton irradiation Similar experiments are now being undertaken using proton beam with energy of 600-800MeV(80-84).

Required technology

The most fruitful field with light ion irradiation is an in-beam mechanical strength test. Determination of creep rate and creep rupture strength are major concerns.

Irradiation creep What is the irradiation creep ? When stress is applied to material at high temperature, deformation increases gradually with time. This phenomenon is called as thermal creep. Irradiation not only accelerates thermal creep rate, but also temperature range at which creep can be seen is extended to much lower temperature. The irradiation creep is one of the important properties in the reactor core design.

What is the requirement for irradiation creep simulation with cyclotron?

Beam stability Irradiation creep rate is proportional to irradiation flux. Creep property is also a strong function of temperature. Thermal expansion disturbs an accurate strain measurement. These factors demand constant flux and temperature in the creep test and it is concluded that,for example, in uni-axial creep apparatus, temperature variation of less than 1 K is desirable to have strain resolution of 10^{-5}.Furthermore, thermal shock and transient effects due to beam fluctuation or beam stop give serious influence on test results. To obtain steady state creep rate, it is necessary to realize a steady state, or at least, quasi-steady state in the point defects production and disappearance processes. Supply of constant and continuous beam is strongly desired. For the compensation of beam heating, direct electric current heating and temperature control system which can respond to beam variation with high speed of time constant of order of milli-seconds is desired.

Table 1 Summary of irradiation creep and radiation damage studies using mainly p and d beam from Cyclotron

First Author	Year	Material	Particle	Energy (MeV)	Dose (dpa)	Dose Rate (dpa/s)	Temperature (K)	Stress Range (MPa)	Main Investigated Item	Ref.
Harkness,S.D.	1971	304	D	22		5x10E-7	773			
Hendrick,P.L.	1976	Ni	P(22),α(70)				497		Description of technology	18
Blanchard,P.	1978	316	α	30	0.7	10E-5	573-723		Temperature dendence,Creep transient	19
Opperman,E.K.	1979	316	P	14.8		3.4x10E-7	673	140	Higher strain rate than neutron	20
Simonen,E.P.	1979	Ni	D			6x10E-7	459-500		Cyclic irradiation	21
Hendrick,P.L.	1979		D	17					Description of apparatus	22
Reiley,T.C.	1980								Description of facility	23
Schwaiger,Chr.	1980	316	P(6.2),D(9)			(1-5)x10E-6	573		Comparison bet. pure and comm. 316	24
Reiley,T.C.	1980	316	α	60						25
Khera,S.K.	1980	FeCrNiMo	D	6		3.3x10E-6		6-100	Void Nucleation	26
Henager,Jr.,C.H.	1980	Ni	D	17			473		Non--linear stress dependency	27
Simonen,E.P.	1981	Ni	D	15		5x10E-7	473		Pulse Irradiation creep	28
Rickerby,D.G.	1981	316					650-900		In-beam fatigue(Preliminary work)	29
Henager,Jr.,C.H.	1981	Ni	D	17		6x10E-7	473	135-250	Stress dependence	30
Riccobono,G.	1982		P,D,He-3,α	10-38					Description of facility	31
Jones,R.H.	1982	Nb	P	16			RT		Tensile properties	32
Jung,P.	1983	Model alloys	P	7			433-673		Stress,temp.,dose rate dependence	33
Jung,P.	1983	316,FeCrNiMo	P	6.2			433-673	250-300	Cyclic irradiation effect	34
Jung,P.	1983	Ni,SS	P	6.2		1.9-2.4x10E-6	573		Stress dependence,Alloying effect	35
Henager,Jr.,C.H.	1983	Ni	D	15-17			473		Weak dependence on initial structure	36
Henager,C.H.	1984	Ni	D	15			473	150	Glide disl. and obstacle interaction	37
Henager,C.H.	1984	316	P		0.21-0.26	3x10E-6	573,693		CIG model	38
Nagakawa,J.	1984	Ni-4Si	P	21		2x10E-7-2x10E-6	623		Cyclic irradiation	39
Bradley,E.R.,		Ni,V,Nb,Ti	P	16			300		Comparison betw. P. and neutron	40
Jung,P.	1986	Ag,Cu,Pt,Ni,etc.	P	6.2					Irradiation creep v. tensile property	41
Jung,P.	1986	Ni-Al	P	6.2					Irr. creep,TEM,Electrical resistance	42
Henager,C.H.,	1986	p.v.steel	P	15			673		Irr. creep,microhardness,TEM	43
With Van de Graaf										
Bemment,Jr.,A.L.	1973	Ni	P	3		6x10E-7	473			
McElroy,R.J.	1976	Ni	P	4-5		1x10E-6	673-823		Dose and dose rate dependence	44
Hudson,J.A.	1977	Ni,321	P	4		1x10E-6	673-873		Stress and temp. dependence	45
Faulkner,D.	1978	Zr,its alloys	P	3.5	0.03		423-623		Irradiation creep and growth	46
Omar,A.M.	1979	Fe,Zr,Cu	P	10-16	-				Electrical Resistivity	47
Lucas,G.E.	1981	Zircaloy2	P	4.75		1x10E-6	628	103-241	Radiation hardened & enhanced creep	48
Atkins,T.	1982	Ni,its alloys	P	3.5	0.2	(0.7-1.7)x10E-6	623-850		Irr. creep,alloying effect	49
Atkins,T.	1986	Ni-1.8Si	P	3.5	0.2		623		Stress effect on defect structure	50

Table 2 Summary of helium implantation tests with cyclotron

First Author	Year	Material	Particle	Energy (MeV)	Helium Cont. (at.ppm)	Inj. Rate (at.ppm/h)	Temperature (K)	Main Investigated Item	Ref.
Ells,C.E.	1963	Al	α	30	0-250			Post He implant. recrystallization	51
Johnson,D.L.	1976	Cu	α	8	200			Site occupancy	52
Charlot,L.A.	1976	Nb	α		30-520			Post He implant. bubble growth	53
Matsumoto,K.	1977		α					Tensile test	54
Snead.Jr.,C.L.	1977	Al	α	50	0.62			Positron annihilation	55
Sagues,A.A.	1978	1.4970	α		150		873-1073	Tensile and creep test	56
Braski,D.N.	1979	FeCrNi	α	28	160		RT	Post He impl. anneal under stress	57
Schroeder,H.	1981	1.4970	α			10-100	1073	In-beam helium creep rupture	58
Sonnenberg,H.	1981	316	α	28	5-3400		673-1223	In-beam and Post He implant.fatigue	59
Shinno,H.	1981	316	α	37	3-10		1023	Post He implant. tensile test	60
Kesternich,W.	1981	1.4970,316	α		100-1000			Post He implant. creep rupture	61
Rothaut,J.	1981	316	α		10-300			Post He implant. bubble growth	62
Abe,K.,	1981	Mo,Mo-Zr	α	10			573	Depth profile of irrad. hardening	63
Schroeder,H.	1983	FeNiCr	α	28	160			Post He implant. creep rupture	64
Rothaut,J.	1983	316	α		10-300			Post He implant. bubble growth	65
Kesternich,W.	1983	1.4970	α		30-1000			Post He implant. bubble trapping	66
Batra,I.S.	1983	316	α	28	5-1000		673-1223	Post He implant. fatigue	67
Schroeder,H.	1983	316,1.4970	α			100		In-beam He implant. creep	68
Kesternich,W.	1984	1.4970,316	α		150		973	Post He implant. creep rupture	69
Kesternich,W.	1985	1.4970						Post He implant. creep test	70
Gadalla,A.	1986	Ni,Cu	α	28	30,000		RT	Microstructure evolution	71,72
Leeser,A.	1986	Ni,Mo,SS	α	32	500		300	Internal friction	73
C.-Filemonowicz,A.	1986	Ni alloys	α	28	500		RT	Microstructure-bubble growth inter.	74
Schroeder,H.	1986	Ni-Si	α	28	0-1000	3.6-108	873-1073	Post impl. creep rupture	75
Packan,N.H.	1986	Ni-Si	P & α	7(P),28(α)	3000appmHe	0.1-0.3dpa	750	Tensile test,TEM,segregation	76
Zhetbaev,A.K.	1986	SS	P & α	30(P),50(α)	10E17P/cm2		190	Mossbauer spectra	77
Ibragimov,Sh.Sh.	1986	Mo	P & α	10-50	0.0001-0.01dpa		323	TEM observation	78
Ibragimov,Sh.Sh.	1986	SS	P & α	7(P),29(α)	500		373	He induced precipitation	79

Beam uniformity Typical specimen dimension in irradiation creep test is 10-20 mm length, 2-4 mm wide and 0.1-0.2 mm thick. Beam uniformity is required to guarantee the uniform damage rate and temperature distribution along specimen length. Usually, mechanical property is strongly influenced by the specimen dimension, especially by the specimen thickness. It is desirable that the specimen contains at least three grains along specimen depth. This means that the specimen thickness is more than 0.1 mm in a usual thermal treatment condition. On the other hand, defect production rate along specimen depth must be uniform. Ion loses its energy and efficiency of defect production increases along its passage. Relative energy loss must be enough small to guarantee uniform defect production rate at beam inlet and outlet. Also, for easiness of the heat removal and temperature control of specimen , specimen thickness must be as thin as possible. To compromise these conflicting conditions, it is concluded that 0.1 to 0.2 mm thickness is a best choice. This choice determines the necessary beam energy which is usually considered to be 10-20 MeV range in case of proton irradiation. So, the application of cyclotron is most popular in this type of experiment.

Beam intensity In simulation test using ion beam, an acceleration test is desirable. This requires high beam current. On the other hand, beam generates heat in the specimen and this heat must be removed efficiently from specimen. Now, beam of several to several tens micro-A/cm^2 is used. This beam generates heat of several tens w/cm^2 in the specimen and the specimen must be forcibly cooled. At the same time, electric current direct heating is necessary to compensate the temperature variation caused by beam fluctuation. The temperature control system mentioned above must be operative under this forced cooling conditions.

Facilities at NRIM

Cyclotron Compact cyclotron manufactured by Japan Steel Works Co. was installed in fiscal year of 1985 at Tsukuba Laboratory in NRIM and preliminary experiments were started on this May. Specifications are as follows.

p	17 MeV	50 micro-A
	4	50
d	10	50
He-3	26	20
He-4	20	20

Computer assisted operation Best operational conditions of ion source(filament current,arc voltage and arc current), magnetic field auxiliary current, dee voltage and deflector voltage are searched at the start and held during experiment with a micro-computer(Hitachi HIDIC-V90-5). Also, beam steering magnet is handled with the computer to hold the beam on the target.

Beam transport To obtain uniform beam profile, two devices are set in the beam line. One is beam scanning device and the other is beam homogenizing magnet. The proton beam can be scanned with frequency of 5 kHz vertically and with 120 Hz horizontally. Fig.3 shows broadening of beam with beam scanning. With this method, ± 10% uniformity was attained along 20 mm specimen length. The beam homogenizing magnet changes beam profile from Gaussian to rectangular distribution. The spacial distribution of beam can be determined with a beam profilemonitor. The beam penetrated through the specimen is measured with a evacuated Faraday cup continuously.

Irradiation creep apparatuses We have two types of irradiation creep apparatuses:torsion type and uni-axial type.These two type machines have peculiar characteristics respectively. The details of performance are given in Table 3.

In one type, the stress is applied tensionally with a dead weight, and in the other, the torsional stress is applied to specimen electro-magnetically.

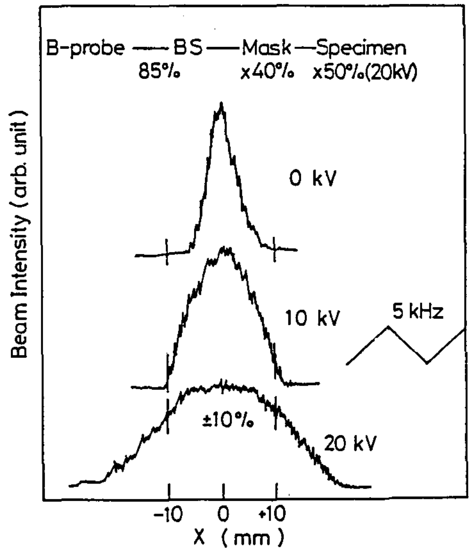

Fig.3 Beam broadening with beam scanning. Wave shape:triangular,frequency:5kHz.

In the torsional type, the rotation angle of wire specimen is measured using an optical lever. Since the thermal expansion does not disturb strain measurement in torsional type apparatus, very high strain resolution of 10^{-7} order can be attained. Also in torsional type test, pure irradiation creep effect can be studied because there is no dilatation stress field and torsion strain is insensitive to void swelling. Some problems are brought about when applied stress goes beyond some limit and irradiation creep rate is not proportional with stress, since the stress distribution is not uniform in the torsional specimen .

In uni-axial tension type test, it is possible to measure large deformation and so, the creep rupture test is easily conducted. Samples for TEM observation can be easily made from this type of test specimens.

The test condition of low damage rate, low temperature and low applied stress is covered with the torsional type apparatus and for the opposite side experimental conditions, the tensile type is appropriate.

Temperature uniformity along specimen length is also essential. Under no irradiation and no helium flow, the temperature of speci men goes higher with higher position of speci men as shown in Fig.4. This is caused by natural convection. This trend is modified with forced convection under helium jet of 100m/s and uniform temperature distribution

Table 3 Comparison of performance between tensile and torsion type irradiation creep apparatusess

Type Item	Tensile	Torsion
Large beam cuurent	possible	possible
High velocity He cooling(100m/s)	possible	possible
Strain resolution	~ 1 x 10^{-5}	~1 x 10^{-7}
Error due to temperature variation	~ 1 x 10^{-5}/deg	~1 x 10^{-7}/deg
Small size of specimen(Low activity)(mm)	20 x 2.5 x 0.1	0.1ϕ x 8
Strain measurement method	LVDT	Optical lever
Dilatation stress field	yes	no
High rate temperature control	desirable	not necessary
Addition of periodic stress cycle	impossible	possible
Observation of creep tested specimen by TEM	possible	difficult
Creep rupture test	possible	impossible
Stress distribution in specimen	uniform(tensile)	non-uniform(torsional)

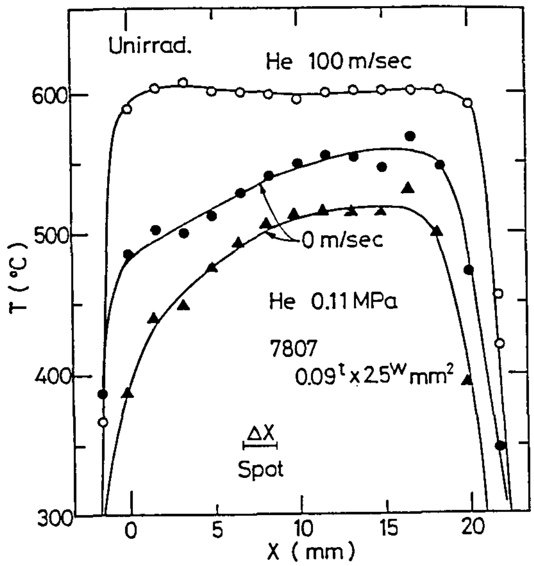

Fig.4 Effect of helium flow rate on temperature profile along specimen length

was obtained.

Some preliminary results at NRIM

In ion beam experiment, stress effect on specimen can be studied only under light ion bombardment.

Irradiation creep test with proton and deuteron is most characteristic and fruitful areas in light ion irradiation technology. Next, the in-beam and post-helium injection creep rupture test have not less importance in the fusion material study. In these mechanical tests, specimen thickness more than 0.1 mm is desirable. Fracture toughness is another important field, but in this case, the specimen thickness more than 1 mm is considered to be necessary and the application of light ion is somewhat questionable. This point must be clarified in future.

Irradiation creep

Figure 5 is the one example of creep test of 316 SS obtained with the torsional apparatus.The remarkable decrease of irradiation creep rate with irradiation time was obtained, as illustrated in Fig.6. The increase of damage rate of factor of 10 caused the decrease of creep rate of factor of about 2-3. The irradiation creep rate was calculated from SIPA model using rate theory and fairly good agreement between calculated and experimental values was obtained, as given in Fig.7.(85-87)

Fig.8 shows the examples of creep curve obtained with the tensile machine. The observed apparent large decrease of elongation was due to the beam stop caused by beam discharge. Fig.9 compares the results of the present work and of KFA-Julich. The difference of beams of proton and deuteron does not make much difference in creep rate. In contrast, the high purity experimental alloys gives much higher creep rate than the commercial 316 SS. Comparison between the NRIM alloy and KFA high

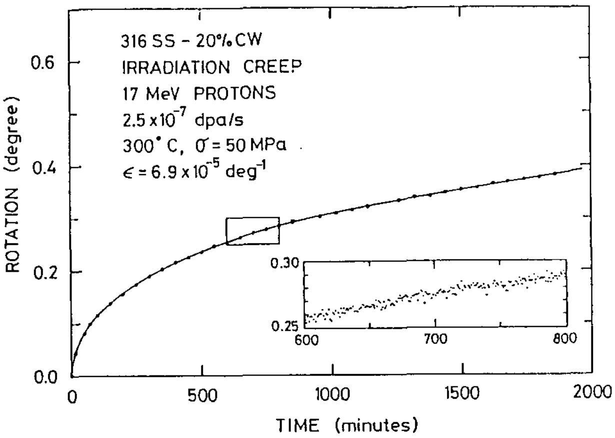

Fig.5 Irradiation creep curve of 20% CW 316 SS under 17 MeV proton bombardment in torsional test.

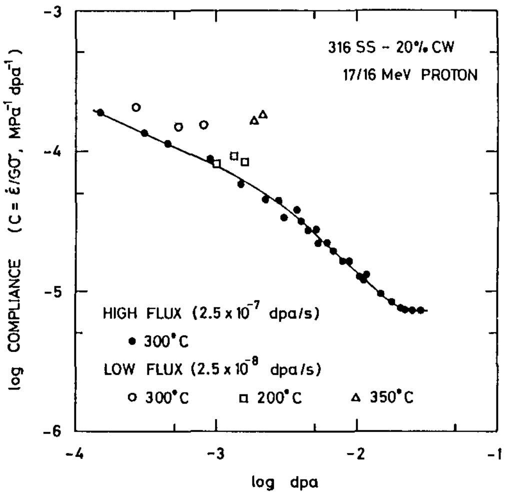

Fig.6 Dependence of creep rate of 20% CW 316 SS on irradiation time and flux.

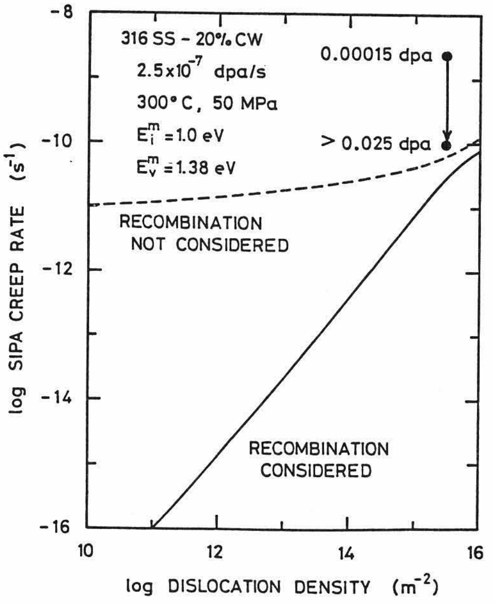

Fig. 7 Comparison of experimental creep rate with calculated SIPA creep rate.

purity 316 SS reveals that higher nickel content does not decrease creep rate. The reduction of creep rate in commercial 316 SS is due to existence of such alloying elements as Si,Mo and Mn.(88)

In initial transition stage, pulsed irradiation caused a shrinkage of specimen length. The details of this phenomenon is now under investigation.

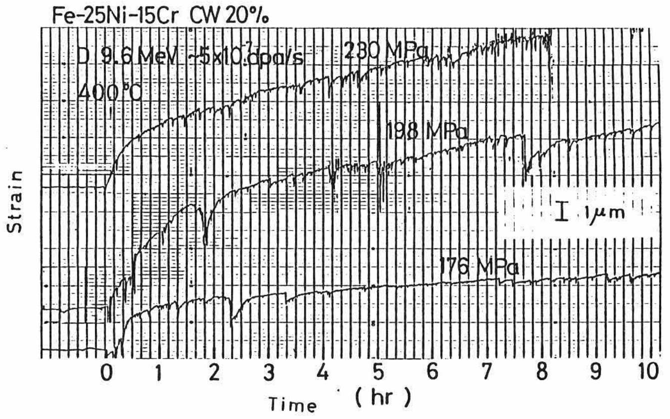

Fig.8 Irradiation creep curve of Fe-25Ni-15Cr experimental alloy under 10 MeV deuteron bombardment in tensile test.

Helium implantation The other interesting field is helium embrittlement. The effect of finely dispersed TiC precipitate was investigated after helium injection at 923 K.

In the solution treated sample(treatment A), only sparse distribution of TiC was seen. In the solution treated and aged specimen (treatment B), fine TiC precipitation was found on grain boundary. In the thermo-mechanically treated condition(treatment C), fine TiC carbide was distributed throughout the matrix uniformly.

The creep curves of these three kind specimens are shown in Fig. 10. The absence of TiC carbide in treatment A caused a premature failure. This premature failure is caused by the existence of grain boundary helium bubbles.

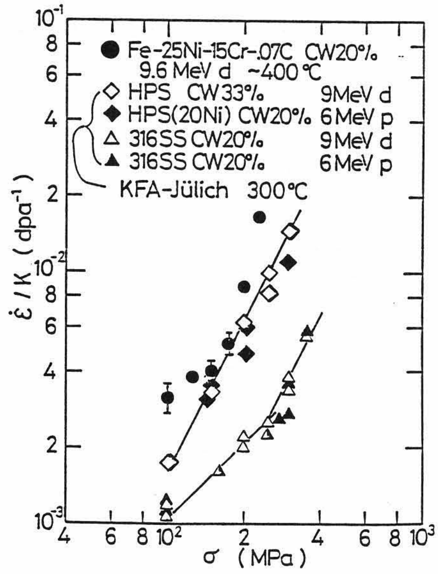

Fig.9 Comparison of irradiation creep data with light ions in NRIM and KFA.

The remarkable improvement can be obtained with the thermo-mechanical treatment: the largest strength and almost no reduction of strength with addition of helium,as illustrated in Fig.11. The details of results are found elsewhere.(89,90)

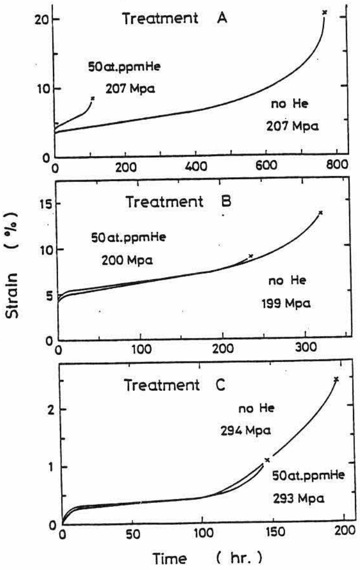

Fig.10 Effect of pre-helium implantation treatments on creep curves of helium implanted PCA alloy.

Future works and conclusion

Further improvement in beam stability,beam uniformity and temperature control is necessary. As high fluence study is also desirable in light ion experiment, the increase of beam intensity and irradiation time is essential. For this object, the computer assisted operation of total experimental system must be advanced further.

In the planned future works, the light ion technology will be applied to basic study and,

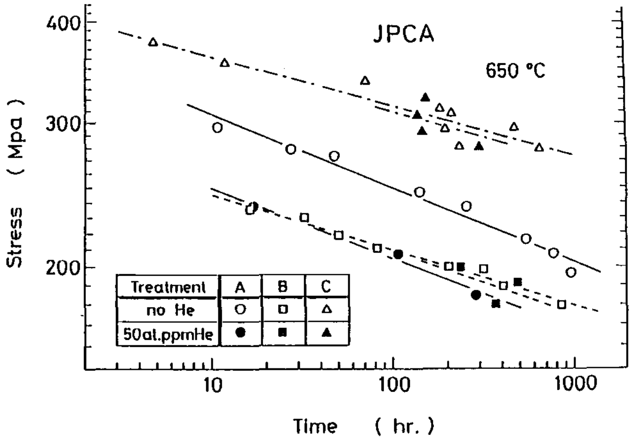

Fig.11 Effect of pre-helium implantation treatments on creep rupture time of helium implanted PCA alloy.

preliminary and screening test of new developmental alloys,as shown in Table 4. One largest problem in this technology is that it is difficult to attain high irradiation dose. To improve this situation, a preconditioning irradiation with fission reactor and a sequential irradiation with cyclotron and reactor will be applicable in future.

Table 4 Planned future works at NRIM.

1. Basic study
 1) Modeling of light ion radiation damage structure development
 2) Irradiation creep mechanism
 3) High concentration helium effect
 4) Establishment of neutron-light ion radiation damage correlation
2. Developmental new alloys
 1) Evaluation of limit and improvement of 316 and PCA type alloys (in view of high concentration He effect)
 2) Screening test of new alloys (monocrystal,oxyde dispersed,rapidly quenched)
3. Development of minisize specimen technology

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