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journal or publication title	Science reports of the Research Institutes, Tohoku University. Ser. A, Physics, chemistry and metallurgy
volume	35
number	2
page range	309-317
year	1991-03-05
URL	http://hdl.handle.net/10097/28350

RADIATION EFFECTS ON METAL MATRIX COMPOSITES BY FISSION NEUTRONS FROM JMTR AND JOYO

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(Received January 9, 1991)

Synopsis

Ceramics fiber reinforced composites are expected to be potential candidates as fusion reactor structural materials. Aluminum matrix composites reinforced with C or SiC fibers are investigated. The objectives of this investigation are to evaluate potential of Al matrix composites as low activation fusion reactor materials and to develop them for fusion applications.

Mechanical properties were measured by three point bending test and mini size specimen tensile test for composite materials and mono-filament tensile test. Microstructure was inspected by means of SEM and TEM. The effects of radiation were studied using fission neutrons of JOYO(FBR) and JMTR(BWR) and 1 MeV electrons from a HVEM. Al matrix composites showed excellent stability under irradiation up to a certain fluence, named threshold fluence. For SiC fibers in the composites, increment of tensile strength and Young's modulus and crystallization of amorphous SiC were observed below the threshold fluence. But at above the threshold fluence, strength drastically dropped. Alloying of Ni and Si to matrix aluminum alloys was suggested to be unfavorable for SiC/Al composite materials used under nuclear reactor environments.

I. INTRODUCTION

Aluminum matrix composite materials are realized to be potential fusion reactor

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materials because of their good mechanical properties at elevated temperatures and low activation characteristics[1,2]. This work has been carried out based on the recent efforts to develop metal matrix composites(MMCs) with Al alloy matrices and SiC fiber or Carbon fiber reinforcement for aeronautic and space applications[3].

Wire	Reinforcement	Matrix	σ_t(GPa)
SiC/Al	PCS-SiC(1800D)	pure Al	1.2
	PCS-SiC(600D)	pure Al	1.4
		Al-5.2%Ni	1.7
		Al-1.7%Ni	1.2
C/Al	M40-S	A1080	1.1
		A5056	1.1
	M40J-S	A1080	1.2

Table 1 List of materials used

The objectives of this investigation are to evaluate potential of Al matrix composite as low activation fusion reactor material and to develop them for fusion applications. The main emphasis of this work is to investigate neutron radiation effects on microstructural evolution and mechanical property changes in Al matrix composite materials. This is a part of the radiation damage study utilizing nuclear reactors, such as JMTR, JOYO and FFTF, 14MeV neutron source, RTNS-II, and dual ion irradiation facility, HIT Facility of the University of Tokyo. In-situ observation of damage process under a high voltage electron microscope (HVEM) is also performed. To evaluate effects of alloying elements in Al alloy matrices and of protection coatings to ceramics (Graphite) fibers on neutron damage tolerance of Al matrix composites is another emphasis of this work. SiC/Al-Ni alloy MMCs have been developed by the authors and been proved their excellent high temperature strength and improved interfacial structures[4].

II. EXPERIMENTAL

The materials used are MMCs with Al alloy matrices, as summarized in Table 1. The Ni and Si contents are ranging 0-7.2 wt.% and 0-2.0 wt.%, respectively. The multi-filament long fibers, PCS-type SiC fibers (Nicalon) and the PAN-type carbon fibers (Torayca) are used as reinforcement. Composite wires used are made with liquid metal infiltration method from the test plants of Nippon Carbon, for SiC/Al, and Toray, for C/Al. The diameters of SiC/Al and C/Al composite wires are 1.0 - 0.3 and 0.5 mm, respectively [5]. The sheet materials of the MMCs are made from the composite wires by hot-press method and hot-rolling method. Mechanical properties are measured by three point bend test and mini size specimen tensile test for composite materials and mono-filament tensile test for reinforcing fibers. The tensile specimens are cut out from the sheet materials. The standard mini-size tensile specimens in the Japanese

Monbusho program is applied to tensile test of composites[6]. Specimen settings for three point bend test and mono-filament tensile test are shown in Fig. 1. Neutron irradiations are carried out utilizing Japanese Materials Testing Reactor(JMTR) and Japanese Fast Breeder Test Reactor (JOYO) at Oarai. In-situ observations of damage process and of microstructural evolution under heat treatments are performed under a 1.25 MeV HVEM, using 1 MeV electrons.

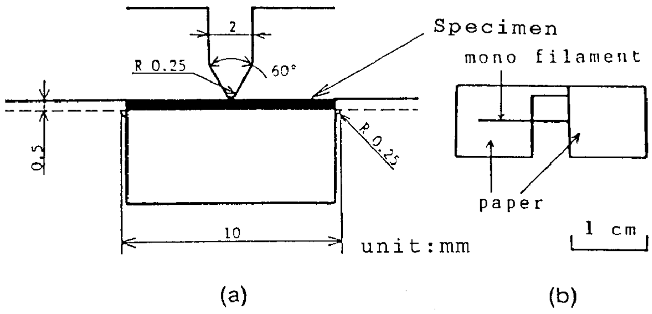

Fig. 1 Schematics of specimen setting (a)three point bend test, (b)mono-filament tensile test

III. RESULTS AND DISCUSSION

(1) Neutron Damage in SiC/A and C/Al

Siliconcarbide fiber reinforced pure aluminum matrix composites, PCS-SiC/pure Al, have been irradiated in JOYO and JMTR with the shape of small tensile test specimen and that of composite wire. Because of limitations on irradiation volume, three point bend test was applied for post irradiation mechanical property test for composites. However, SiC fibers were tensile tested, as three point bend test was not feasible for the fine fibers. To correlate these two kinds of test results, the correlation rule between flexural strength and tensile strength was introduced using a data set of unirradiated composites, as shown in Fig.2. From the data set, the following equation is derived as,

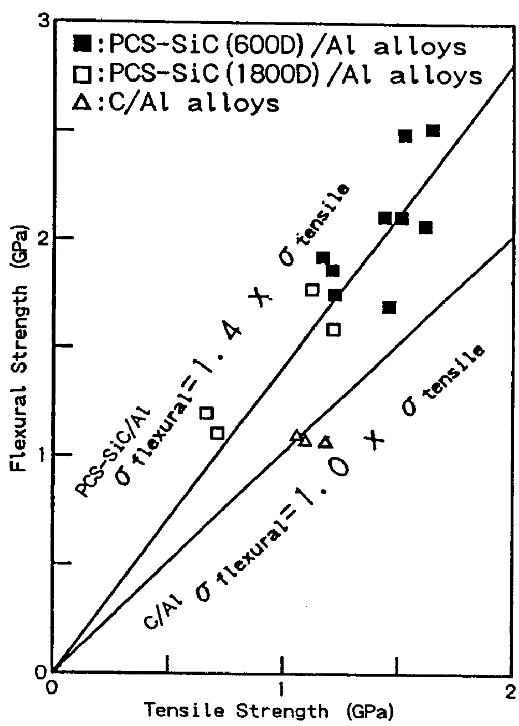

Fig. 2 Correlation between tensile strength and flexural strength for C/Al and SiC/Al

[flexural strength] = α x [tensile strength] (1)

where the value of α is 1.4 and 1.0 for SiC/Al and C/Al, respectively. The higher α value for SiC/Al than for C/Al can be attributed to the higher interfacial bonding strength between fiber and matrix for the case of SiC/Al[6].

Figure 3 shows neutron fluence dependence of flexural strength and tensile strength for SiC/Al and extracted SiC fibers. In the figure, flexural strength for composites and tensile strength for extracted fibers are empirical values. Using the correlation equation (1), an theoretical strength based on a simple rule of mixture can be obtained with a fiber strength and a fiber volume fraction value. The calculated value coincided well with the experimentally obtained value up to the irradiation dose of 1 x 10^{25}n/m^2. This is suggesting that neutron irradiation does not affect interfacial bonding characteristics up to the dose. Neutron dose dependence of the irradiation strengthening is shown in Fig. 4. Radiation hardening in SiC/Al is indicated to be proportional to the (neutron dose)$^{1/3}$. This neutron fluence dependence is similar to the irradiation hardening due to dispersion hardening by radiation induced defect clusters with three dimensional uniform distribution. This can be related to the microcrystal formation in the amorphous SiC fibers. At the highest irradiation dose in JOYO extracted SiC fibers become undurable for tensile test which corresponds to a drop of composite strength. Surface morphology of the extracted fibers after neutron irradiation to 9 x 10^{25}n/m^2 is shown in Fig. 5 in comparison with that of the unirradiated fibers. Neutron irradiation at 723K for 9 x 10^{25}n/m^2 results in the radiation induced surface roughing due to the interfacial reaction between fiber and matrix. This can be another reason why the composite strength drops at the irradiation dose. Fiber strength extracted from composites

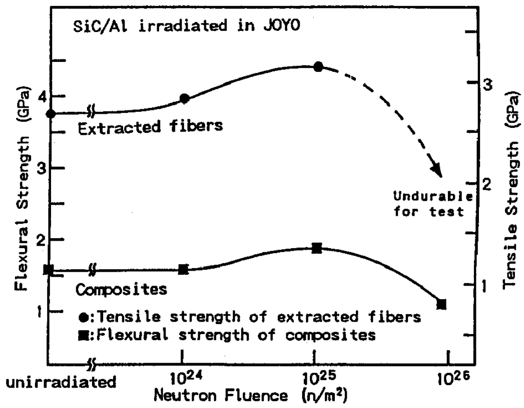

Fig. 3 Neutron fluence dependence of flexural strength for SiC/Al composite irradiated in JOYO

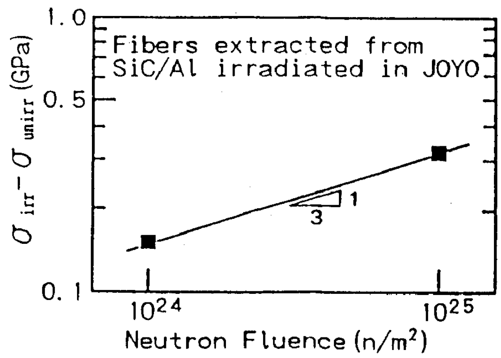

Fig. 4 Neutron fluence dependence of irradiation strengthening

is examined for unirradiated and irradiated conditions and is indicated as relative strength, strength of irradiated fibers divided by strength of unirradiated fibers, in Fig. 6. Both for JMTR and JOYO irradiation, relative strength increases at an early part of the irradiation, that is at lower neutron dose. Then after the peak dose with the peak strength for each irradiation, relative strength steeply decreases with increasing neutron dose. The peak dose is about one order of magnitude smaller for JMTR irradiation than for the JOYO irradiation and the relative strength at the peak dose is about 30% higher for the former than the latter. The major difference in the irradiation condition is the neutron energy spectra, neutron flux and irradiation temperature. Since crystallization temperature for PCS-SiC is about 1800K, the temperature difference between 300K and 723K may not be a major origin of the above mentioned radiation response. The differences in displacement damage rate, H and He gas generation rates can be the origin to produce the different behaviors under neutron irradiation. X-ray diffraction analysis, Fig. 7, suggests micro-crystallization of amorphous SiC. This crystallization behavior is also observed by dual-ion irradiation of SiC/Al composite

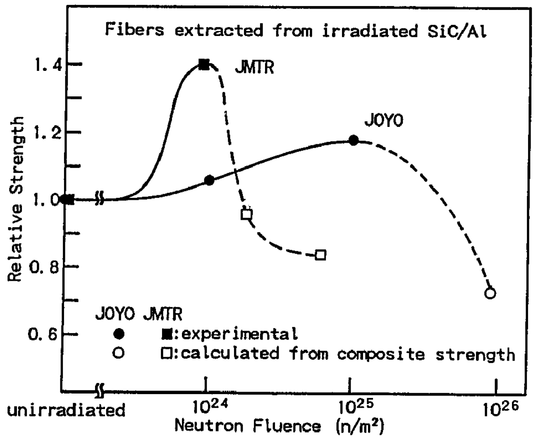

Fig. 6 Neutron fluence dependence of SiC fiber strength irradiated in JOYO and JMTR

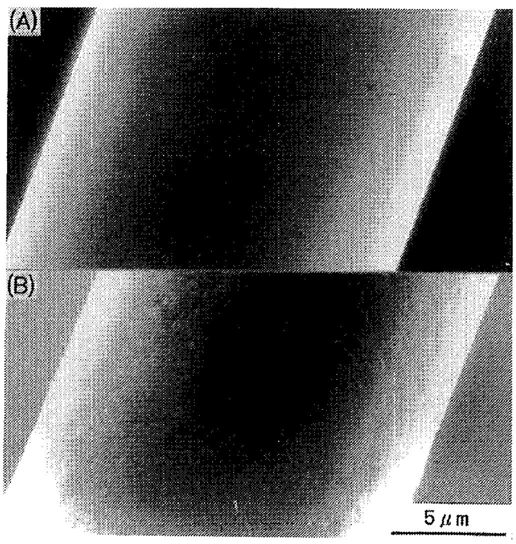

Fig. 5 Surface morphologies of SiC fibers extracted from SiC/Al (A)un-irradiated, (B)irradiated to 9.0 x 10^{25}n/m^2 at 723K in JOYO

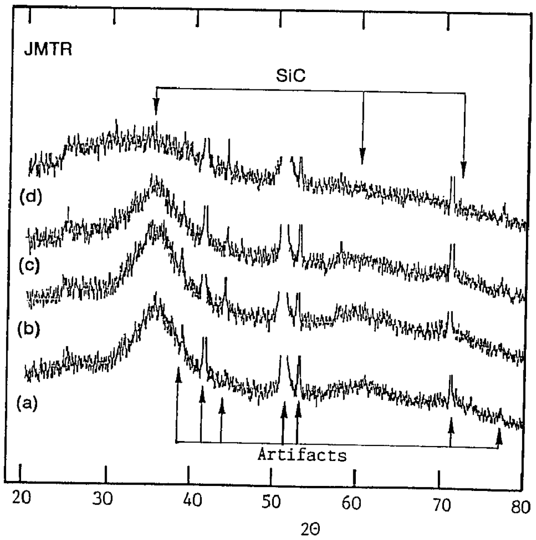

Fig. 7 Effect of neutron irradiation on microstructure of SiC fibers (XRD analysis)

microcrystals than that observed in thermally crystallized materials[3]. Radiation induced crystallization is also reported as the drastic shape change in the same SiC/Al by the cycle 10 irradiation of FFTF/MOTA[7].

C/Al composites are irradiated in JMTR. PAN-type carbon fibers are simultaneous Ti-B CVD coated prior to liquid metal infiltration process. The important factor to decide the composite strength is a soundness of interfacial protection layers. This layers act to protect interfacial reaction and to enhance wettability of Al matrix to fibers, but they provide a weak interfacial bonding strength. Neutron irradiation to C/Al composites seem to change interfacial microstructure to increase bonding strength between matrix and protection layers. This is suggested by fracture surfaces of the composites. Figure 8 provides SEM fractographs of M40J-S/A1080 before and after neutron irradiation to $1.8 \times 10^{24} n/m^2$ in JMTR. Before the irradiation, C/Al composites have many pull-outs proving that they possess good interfacial structure. Whereas after the irradiation, pull-outs is suppressed indicating a smooth crack propagation from fiber to matrix. Fraction of the strength drop for the three composites, M40J-S/A1080, M40S/A1080 and M40J-S/A5056, is 22 to 24% from the strength before the irradiation. This radiation induced mechanical property degradation takes place more than one order of magnitude lower than that for the SiC/Al. High fluence neutron irradiation in the cycle 10 of FFTF/MOTA indicates definitely smaller dimensional change for C/Al than for SiC/Al[7]. As a guideline to develop MMCs with higher neutron damage tolerance, importance of

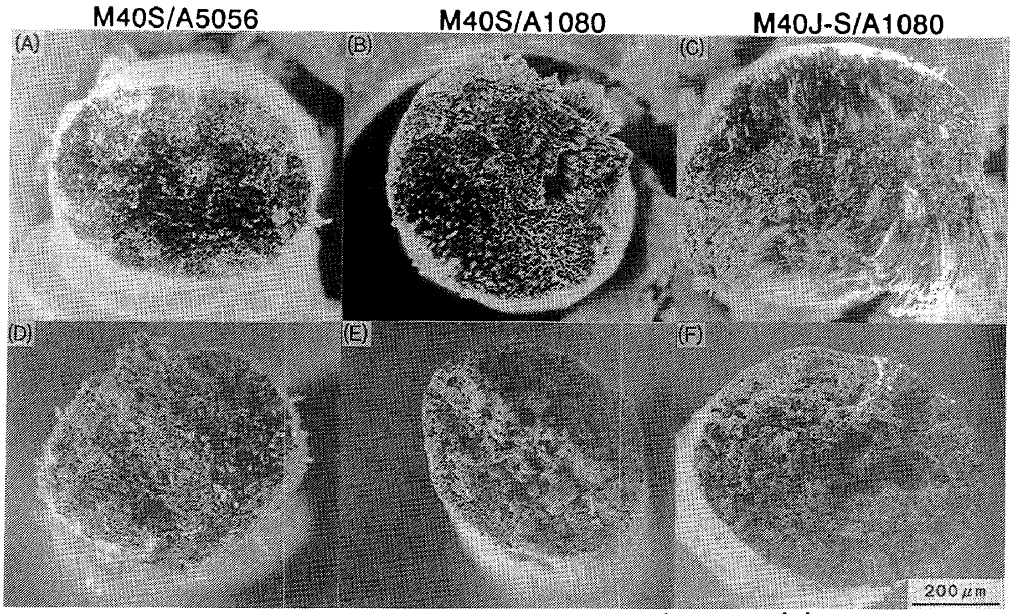

Fig. 8 SEM fractographs of C/Al-alloy composite materials
(A),(B),(C):un-irradiated
(D),(E),(F):irradiated to $1.8 \times 10^{24} n/m^2$ at 723K in JMTR

crystallization control for PCS-SiC/Al and of interfacial reaction control for C/Al can be highlighted.

(2) Impacts of Ni and Si Addition to SiC/Al-Alloy

Nickel addition to Al matrix has been introduced to improve high temperature strength of SiC/Al composite materials[5]. Silicon addition was also expected to

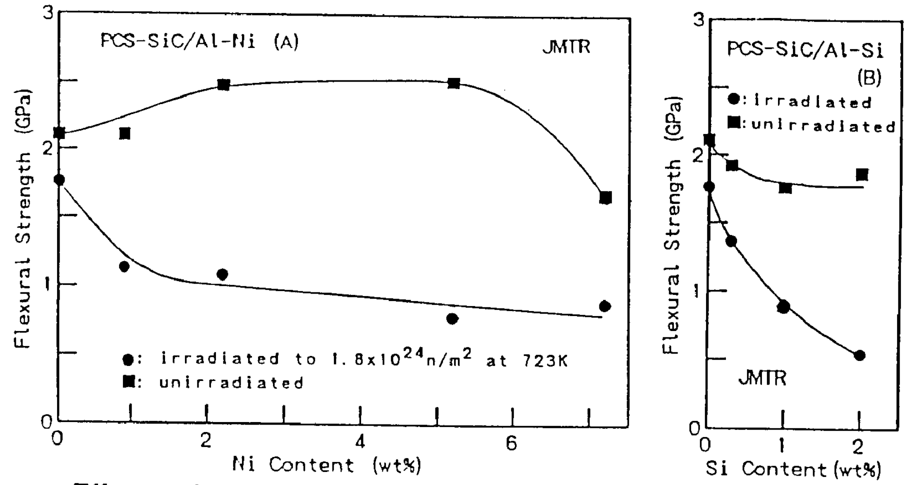

Fig. 9 Effects of alloying elements in Al alloys on flexural strengths of SiC/Al (A)Al-Ni and (B) Al-Si matrices

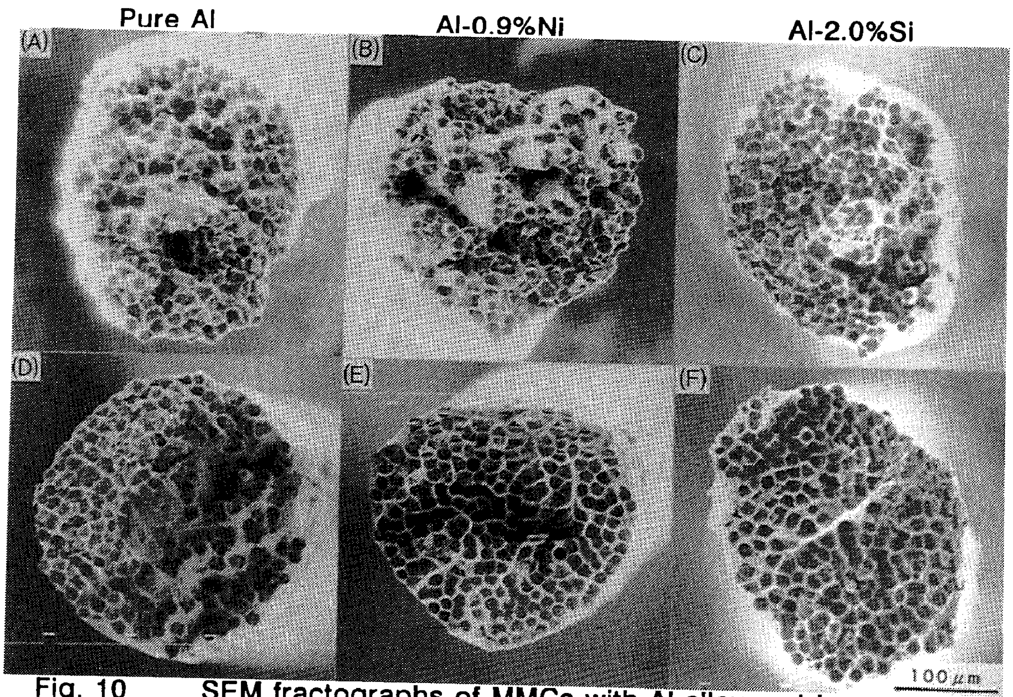

Fig. 10 SEM fractographs of MMCs with Al-alloy matrices
(A),(B),(C):un-irradiated
(D),(E),(F):irradiated to 1.8 x 10^{24}n/m^2 at 723K in JMTR

improve characteristics of SiC/Al through an improvement of matrix strength, but hitherto, no improvement effects have been observed as to mechanical properties. These two matrix alloy systems have been subjected to neutron irradiation in JMTR. Figure 9 provides the effects of Ni and Si addition to Al alloys on flexural strength. With increasing Ni, strength of composite gradually increases up to 5.2% Ni addition. This Ni concentration corresponds to the fully eutectic structure of Al-Al_3Ni and excess addition of Ni causes large drop of flexural strength. Neutron irradiations to the Al-Ni alloys are found to be detrimental to mechanical properties. The drastic drop of the flexural strength to 1 GPa is taken place with the Ni addition of 0.9 % and the further Ni addition up to 7.2% leads to gradual degradation. In Fig. 10, typical fracture surfaces for Al-Ni matrices are indicated in comparison with before and after irradiation. Those composites are excellent in strength with high bonding strength between matrix and fibers and result in no pull-outs with step wise surfaces. While neutron irradiation produces catastrophic crack propagation with smooth surface. This should be connected with the microstructural change in matrix by neutron irradiation. That is, radiation enhanced spherotization of Al_3Ni in Al-Al_3Ni eutectic structure may loose crackarrest properties of the eutectic structure. In-situ observation of damage process in Al-Ni matrix is shown in Fig. 11. The mixed lamellar structure observed before irradiation changed into spherotized structure by electron irradiation. Silicon addition degrades strength slightly about 10% of the strength without Si addition up to the maximum concentration of 2%. Neutron irradiations to the Al-Si alloy matrices are realized to be unsafe for loosing strength steeply with increasing Si. The gradual decrease in strength may correspond to interfacial reactions enhanced with Si. The mechanism of microstructural or microchemical evolutions by neutron irradiation is under investigation and the precise analysis of the phenomena would open the gateway to the newadvanced MMCs for fusion.

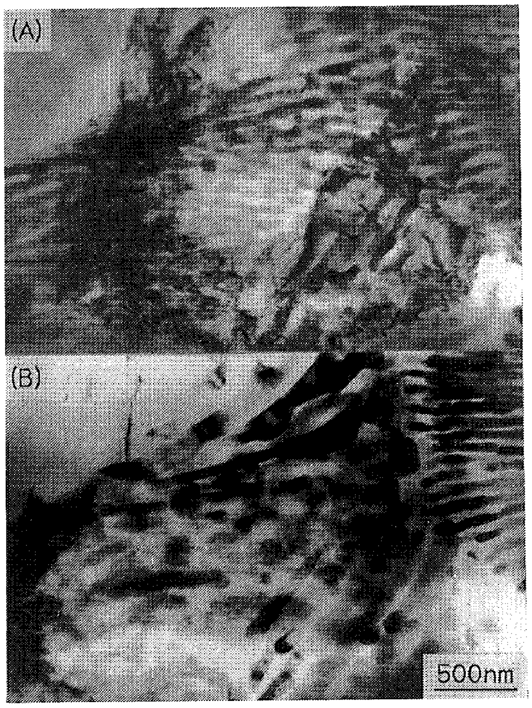

Fig. 11 Spherotization of Al_3Ni in PCS-SiC/Al-5.2%Ni composite wire under HVEM with 1 MeV electron bombardment for 60 min. at 573K
(A)un-irradiated,
(B)irradiated

IV. CONCLUSIONS

[1] Effects of neutron irradiation on metal matrix composites were studied utilizing fission reactors; JOYO(FBR) and JMTR(BWR). Al matrix composites showed excellent stability under neutron irradiation up to a certain fluence, named threshold fluence.
[2] For SiC fibers in the composites, increment of tensile strength and Young's modulus and crystallization of amorphous SiC was observed below the threshold fluence. At above it, strength drastically dropped, both for C/Al and SiC/Al.
[3] The threshold fluence for SiC/Al is about one order of magnitude higher than that for C/Al. For the case of C/Al, radiation enhanced interfacial reaction was interpreted to be the origin of the degradation.
[4] Nickel and silicon alloying to Al are proved to be detrimental for durability to neutron irradiation.

Acknowledgement

The authors would like to express their appreciations to Drs. Y.Imai of Nippon Carbon Co. Ltd., T.Kyono of Toray Co. Ltd. and A.Sakamoto of Mitsubishi Heavy Industries. Sincere encouragements and fruitful discussions to this work by Dr. Y.Minoda, Prof. N.Igata and Prof. A.Kobayashi are acknowledged.

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