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# Development of Controlled Temperature-Cycle Irradiation Technique in JMTR*

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The effects of cyclic temperature changes during neutron irradiation upon radiation induced microstructure evolution and resulting property changes of materials is very important from both fundamental and engineering viewpoints. Therefore, a technique that allows us to do the controlled temperature-cycle irradiation was developed in the Japan Materials Testing Reactor (JMTR). The technique meets the following requirements: (1) the temperature-cycle irradiation is to be performed under three different conditions by changing lower and upper temperatures; 200 --- 400°C, 300 --- 400°C and 300 --- 450°C. (2) the number and period of the temperature-cycles are to be six for 24-day full irradiation and approximately 44 h/44 h at the lower/upper temperatures. (3) the temperatures of each specimen assembly are to be maintained at the lower temperatures before start-up of the reactor and at the upper temperatures during shut-down until the complete absence of reactor power. In this paper, the details of the irradiation rig, successful results and several problems to be overcome for future improvement are presented.

KEYWORDS: neutron irradiation, temperature cycle, JMTR, irradiation rig

## 1. Introduction

Is it possible to predict the effect of irradiation temperature variation that inevitably occurs after the start-up of currently operating fission and future fusion reactors upon radiation induced microstructure evolution and resulting property changes of materials from the current understanding based on the constant temperature irradiation conducted so far ? The answer will be "*No*", since there are no available reports on this effect. This is due mainly to the lack of the technique that allows us to do controlled temperature-cycle irradiation. Therefore, we have made efforts for the development of such a technique since 1990 in the Japan Materials Testing Reactor (JMTR) at the Oarai Research Establishment of Japan Atomic Energy Research Institute. The JMTR, one of light water reactors with a full thermal power of 50 MW, was specially designed for irradiation testing of nuclear materials and fuels, attained the first criticality in 1968 and now is well known to be one of the most advanced materials testing reactors in the world. At first, we designed an in-core irradiation rig (or capsule) that permits both the temperature-cycle irradiation under three different conditions and temperature-constant irradiation, the rig being divided into four sections for independent temperature control. The result of our trial, however, was not satisfactory because of the occurrence of uncontrolled temperature variations due mainly to strong thermal interaction between the four sections. In view of this experience, we decided to limit the irradiation conditions to only the temperature-cycle irradiations of three different temperature histories and designed an improved in-core irradiation rig in 1991. As a result, we have confirmed the completion of development of the technique. The present paper will present the details of the in-core irradiation rig, successful results and several problems to be overcome for future improvement.

## 2. In-core irradiation rig

The in-core irradiation rig to be developed meets the following requirements.

(1) The temperature-cycle irradiations are to be performed using a single capsule under three different conditions by changing lower and upper temperatures; 200 ↔ 400°C, 300 ↔ 400°C and 300 ↔ 450°C, in an irradiation hole in the JMTR reactor core which provides the total dose of fast neutron (E >1 MeV) approximately equal to 1.4 x $10^{24}$ n/m$^2$. (2) The number and period of the temperature-cycles are to be six for 24-day full irradiation and approximately 44 h/44 h at the lower/upper temperatures, respectively. (3) The increasing rate of temperature between the lower and upper temperatures is to be as fast as possible, and the decreasing rate is to rely on cooling by just cutting off heating currents. (4) Temperature measurements are to be made at three positions for each of three divided sections inside the rig to assess the irradiation temperature of specimens concerned by knowing the temperature distribution along the longitudinal and radial directions of each section. (5) The temperatures of each specimen assembly are to be maintained at the lower temperatures before start-up of JMTR and at the upper temperatures during shut-down until the complete absence of reactor power. This is necessary to eliminate the specimen exposure to neutrons at uncontrolled varying temperatures during the start-up and shut-down [1]. (6) Four sets of specimen assembly to be accommodated in each of the three sections.

The schematic of the planned temperature-cycle irradiation experiment which meets the above items of (1), (2), (3) and (5) is shown in Fig. 1. The irradiation hole used in this study was J-5 (Fig. 2), which has a fairly high neutron flux of 5.9 x $10^{17}$n/m$^2$s$^1$, resulting in total neutron dose of 1.4 x $10^{24}$n/m$^2$ (E>1 MeV) for 24-day full irradiation [2, 3]. Figure 3 shows the γ-ray intensity distribution along the longitudinal direction in J-5 and the

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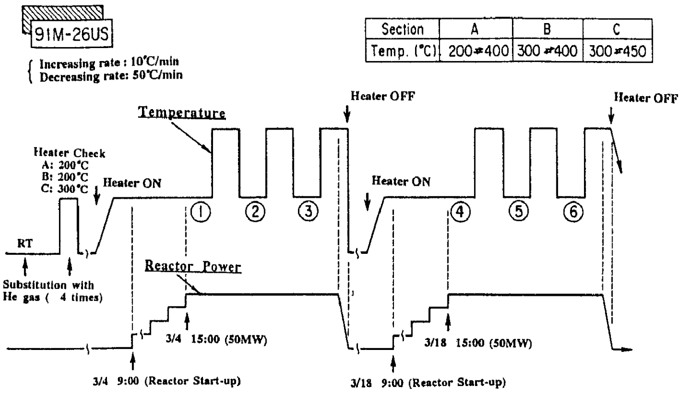

Fig. 1 Schematic of the planned temperature history before, during and after reactor power operation in the controlled temperature-cycle irradiation experiment.

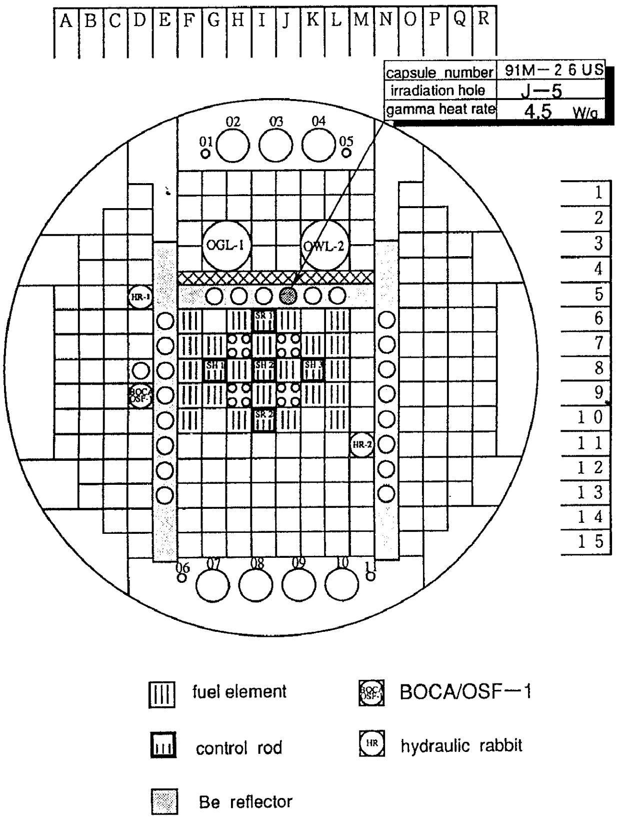

Fig. 2 Irradiation hole of capsul in JMTR.

location of specimen assemblies in three divided sections, A, B, and C. After the start-up of reactor, especially after the full power (50 MW) operation of reactor, heating of specimens by γ-rays occurs significantly and it increases with increasing neutron or γ-ray flux. Therefore, irradiation temperatures for the top, middle and bottom sections in Fig. 3 were chosen to be 200↔400°C, 300↔400°C and 300↔450°C, respectively.

Figure 4 gives the vertical and transverse cross-sections of the in-core irradiation rig. The inside of the rig was divided into three sections, A, B and C, in each of which four sets of specimen assembly are accommodated and the independent control of temperature can be performed. In order to know the irradiation temperature distribution inside the individual sections, three thermocouples were

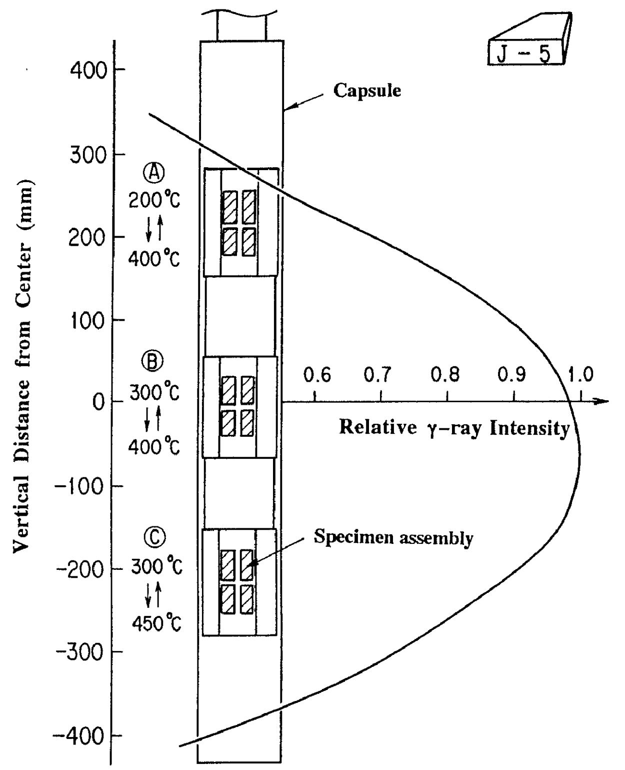

Fig. 3 Vertical distribution of relative g-ray intensity in the irradiation hole of J-5 and the location of specimen assemblies in three divided sections, A, B, and C.

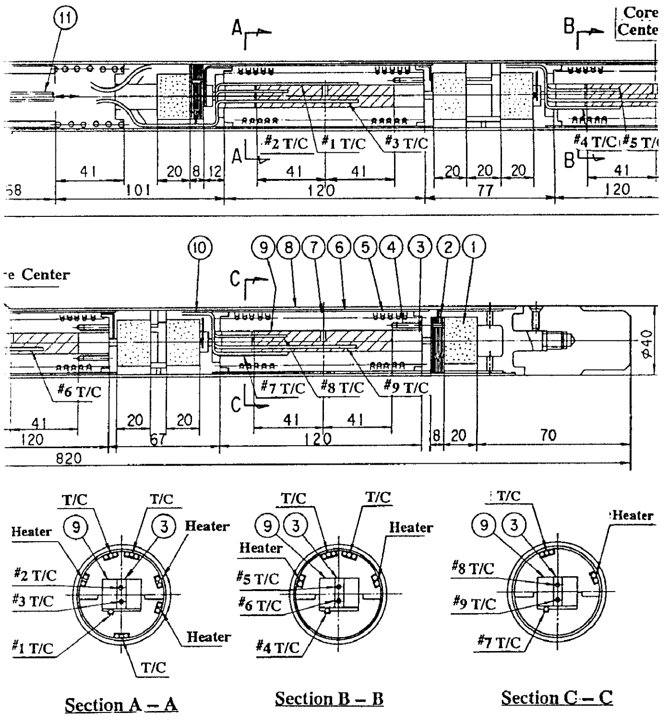

Fig. 4 Vertical and transverse cross sections of the irradiation rig specially designed for the present controlled temperature-cycle irradiation experiment in JMTR. 1: insulator, 2: reflector, 3: spacer, 4: fluence monitor, 5: electric heater, 6: reflection tube, 7: thermal bond, 8: outer tube, 9: specimen assembly, 10: thermocouple, 11: helium gas supplying tube.

placed in each section. For the temperature control, thermocouples marked by #2, 5 and 8 were employed. Interior of the rig was substituted with high purity (6N) helium gas by the repeated exhausting of air with a vacuum pump and introducing of the gas, using tubes for vacuum control and helium gas supply. Since the thermal conductivity of helium, which is best of gases, is three order lower than metals, the controlling of lower temperatures, especially the lowest temperature of 200°C, depends on the degree of realization of intimate thermal contact between specimens and cooling water surrounding the rig. Thus, great efforts to minimize the helium gas gap existing inside the rig were made. For thermal bond in the rig, solid aluminum alloy, A1050, which has a good thermal conductivity and less γ-heating was used. In addition, in order to prevent thermal interaction between neighboring sections, which was essential to do the independent controlling successfully, two pieces of insulating substances of 20 mm thick mullite were used.

A specimen assembly was made of elongated cube shaped vacuum-tight copper box [4]. In the box with dimensions of about 6 mm x12 mm x 40 mm, TEM and mini-tensile specimens, as seen in Fig. 5, of various kinds of materials, such as ferritic and austenitic steels, vanadium alloys, copper alloys and molybdenum alloys, which had been wrapped with high purity aluminum foils, were tightly packed in holes in a copper block for good thermal contact. The holes were machined to take almost the same two-dimensional shapes as those specimen shapes. In order to minimize the oxidation of the specimens due to degassing during the irradiation, inside of each box with specimens was exhausted to a vacuum of less than 2 x $10^{-6}$ torr, then heated to about 150°C and finally mechanically vacuum tightened. To ensure the mechanical tightening, fully annealed oxygen free copper box was used.

## 3. Results and discussion

Figure 6 shows the record of temperature vs time (here date) curves together with reactor power and control rod position vs time curves during the irradiation. Serrations seen in Fig. 6(a) resulted from putting in and out the

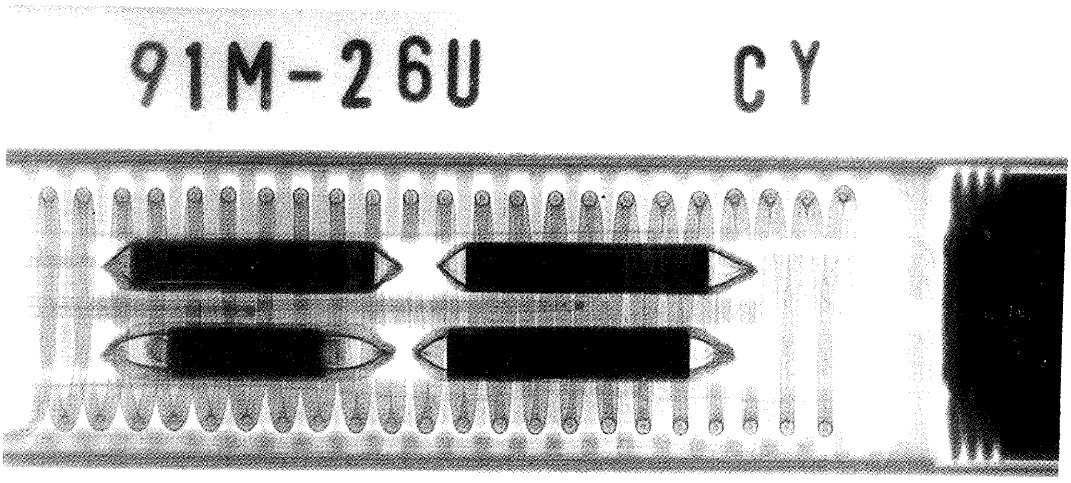

Fig. 5 X-ray photograph showing the interior of in-core irradiation rig with specimen assemblies in section C.

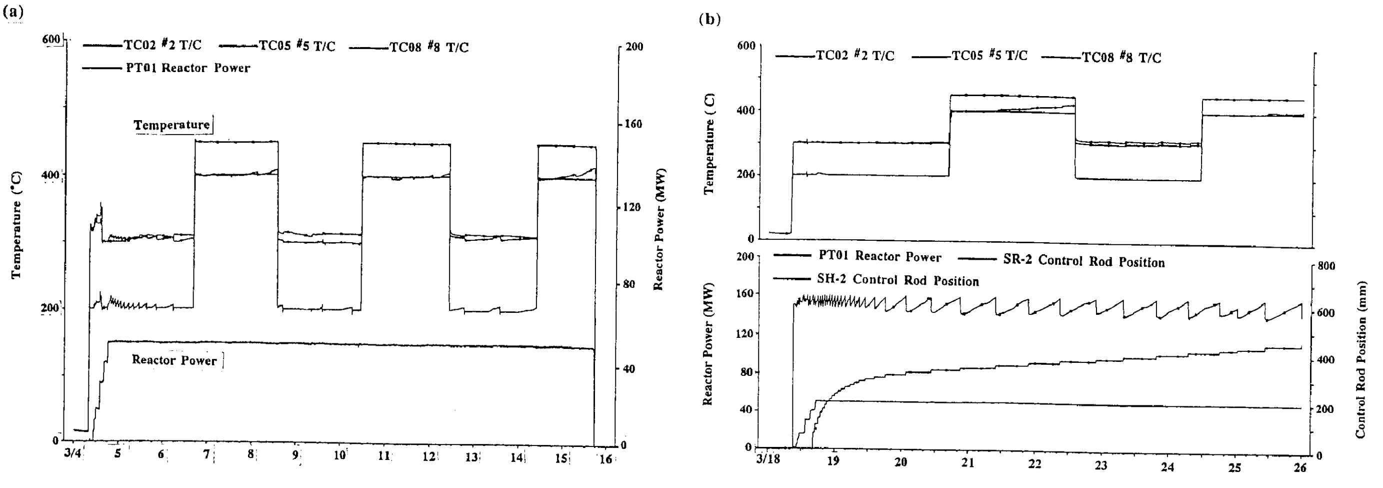

Fig. 6 Records of temperature indicated by thermocouples of #2 (section A), #5 (section B) and #8 (section C) used for temperature control of each section during the irradiation together with the output of JMTR thermal power. (a) the first half, (b) the latter half.

control rods and thus are clearer with decreasing irradiation temperature. This is due simply to the decreasing of heating current. It should be noted from the figure that the recorded temperature vs time curves show a good accordance with the designed curves in Fig. 1. The lowest temperature of 200°C is also seen to be essentially realized. For the present success of controlled temperature-cycle irradiation the following points should be noted : (1) For the temperature control, helium gas pressure control due to combined utilization of both the cooling effect of helium gas and the less cooling effect of vacuum was very effective as well as the heating current control. The combined use under full power operation of JMTR was first tried in our temperature-cycle irradiation experiments. (2) Negligible interaction between sections, A, B and C, was performed by using thick insulators and spacers of less γ-heating.

On the other hand, several problems were encountered. One is the uncertainty of the irradiation temperatures measured with thermocouples. The recorded maximum temperature was 450°C. Nevertheless, aluminum foils used for wrapping TEM and mini-tensile specimens did not exist after irradiation in many cases. According to equilibrium phase diagrams [5], copper and aluminum form an eutectic alloy at approximately 17 at% aluminum with melting point of 548°C, which is about 110°C lower than that of pure aluminum. It is thus probable that the actual specimen maximum temperatures were at least 100°C higher than the recorded ones, though such temperature increase might have occurred instantaneously or for a short time. As mentioned previously, the inside of the box was kept in vacuum to protect pre-thinned specimens from oxidation and each thermocouple was arranged to touch with the outer surface of each copper block (see Fig. 4). If thermal contact of specimens with copper block is maintained through the irradiation, temperature difference between the recorded and actual temperatures may be insignificant. Thus, the observed temperature difference as large as 100°C suggests that thermal contact of specimens with copper block was not always maintained during the irradiation. Thermal expansion coefficient of copper is larger than those of most specimens, thereby increasing the gap between them as the irradiation temperature is increased. As long as good thermal contact is not ensured, the box must be filled with pure helium to increase the degree of thermal contact. In any case, it is necessary to develop a better technique for measuring the actual specimen temperatures.

The irradiation temperatures indicated by three thermocouples in each of sections are shown in Fig. 7. It is obvious that the thermocouples placed in the position closest to cooling water, i. e., #1, 4, 7 (see Fig. 4), indicate lower temperatures than the others attached to the central positions between specimen assemblies. Especially for #4 and #5 which have the same longitudinal positions each other, the observed temperature difference is as large as 70°C, indicating the presence of a large temperature gradient along radial directions. On the other hand, temperature difference along longitudinal directions is not significant.

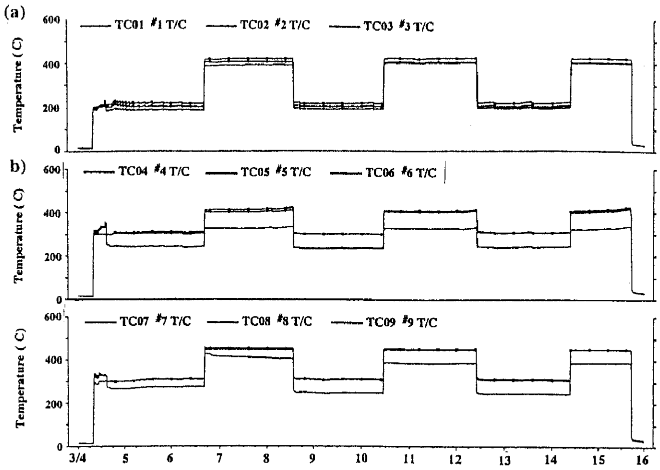

Fig. 7 Records of irradiation temperature showing the temperature distribution inside each of the three divided sections. (a), (b) and (c) correspond to section A, B and C, respectively.

## 4. Conclusions

A technique to perform the controlled temperature-cycle irradiation was at first developed in the JMTR. The technique required the helium gas pressure control due to combined utilization of both the cooling effect of helium gas and the less cooling effect of vacuum under full power operation of JMTR as well as the heating current control for the aimed temperature control. The next step to be made for future improvements of the technique is to establish the method for measuring the actual specimen temperature precisely.

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