

Technique for cladding of poison sheets for Cd-poisoned moderator

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Abstract

To make use of cadmium (Cd) as a poison structure in the liquid hydrogen moderator, various cladding methods such as explosion, heating up a solder of 70%Cd–30%Zn in Al case, cold spraying and cold rolling as well as HIP method were tested to seek the best fabrication method. Cd could be partially bonded to aluminum (Al) in all cases. The explosion method made a rather rigid bond between Al and Cd but generated a crack on Al plates behind Cd plate. Solder also bonded well with the Al case, although a part of the Al case was dissolved due to a mixture of Zn and Al. A porous Al layer on Cd plate was formed in the cold spray treatment. The applicability of the ultrasonic test is also discussed for the non-destructive defect inspection of the bonded materials.

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1. Introduction

The Japanese Spallation Neutron Source, JSNS [1] is one of the worldwide intense pulsed neutron sources and is now being constructed under the JAERI–KEK Joint Project of the Intense Proton Accelerator Facility whose

acronym is J-PARC (Japan Proton Accelerator Research Complex) [2]. The purpose of the installation is to carry out research on material structures and life-science by means of neutron scattering methods. The TMRA (i.e., target-moderator-reflector assembly) of JSNS is composed of a mercury target, liquid hydrogen moderators, and composite reflectors of beryllium and iron as shown in Fig. 1. It has 23 neutron beam lines for neutron spectrometers.

For neutron scattering experiment using the time-of-flight method, moderator performance is characterized by the pulse characteristics: intensity, pulse width

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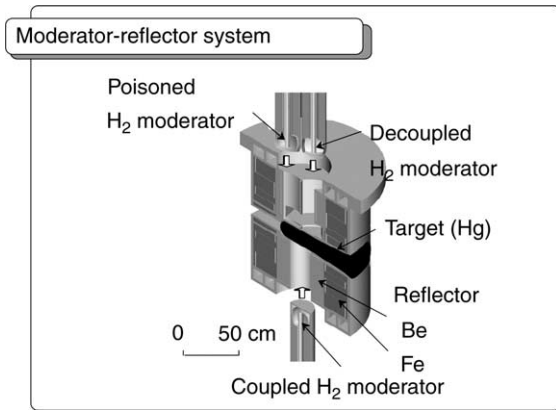


Fig. 1. Target-Moderator-Reflector Assembly of JSNS.

(FWHM, full width at half maximum) and its decay time. The moderators of JSNS were designed to provide a high performance as follows [3–9]. Three kinds of liquid hydrogen moderators (i.e., coupled, decoupled and poisoned moderators) are adopted for the individual research objectives. Especially, the poisoned moderator is required to produce neutron pulses with narrow width for high resolution neutron scattering experiments (e.g., material dynamics). The poisoned moderator system consists of four layers of aluminum (Al) alloy containers, hydrogen (20 K), vacuum, helium (He) and of cooling water as shown in Fig. 2. The thermal neutron absorbing materials (so called ‘decoupler’ and ‘poison’) were also adopted to obtain narrow neutron beam pulses. The decoupler is arranged in Al alloy between the He and cooling water layers except for the neutron beam extraction surface. Higher decoupling energy is very effective for reducing decay time. Boron carbide (B_4C) is already utilized for this purpose in KENS

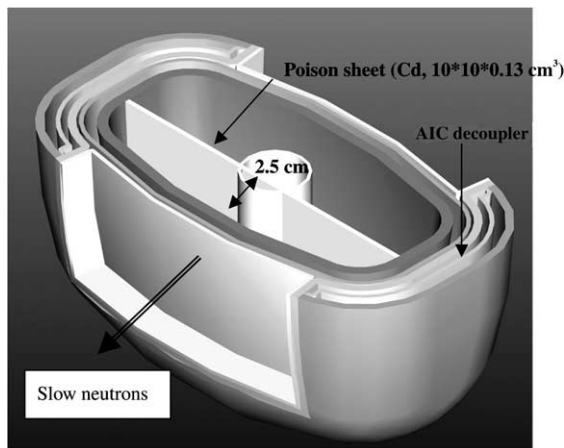


Fig. 2. The poisoned moderator.

[10], IPNS [11] and ISIS [12]. However, He gas generation due to $^{10}B(n, \alpha)$ reaction shortens the decoupler life time in MW class system. In JSNS, new material of Ag–In–Cd alloys with a high decoupling energy of about 1 eV is developed to provide a long life-time usage even in the very intense neutron field.

A poison [13] brings about narrow pulse widths which are controlled by moderator thickness. The poison sheet is inserted in a moderator chamber to control the effective moderator thickness and obtain additional intensity gain from the back side of the moderator divided by the poison sheet. Gd was widely utilized for a poison material in various pulsed neutron sources (ISIS, IPNS, KENS [14,15], etc.) However, it has many resonances above 1 eV and they reduce the slow neutron intensity. On the other hand, the neutron capture cross-section of Cd is very low above 0.4 eV. Neutronic calculation [5] of JSNS showed that a shape of neutron pulse from a Cd-poisoned moderator near the cut-off energy was better than that of a Gd-poisoned system, because Cd cross-section has nearly step-wise change at the cut-off energy, moreover because the cut-off energy of Cd is higher than that of Gd.

The design requirements for a Cd poison are as follows:

- (1) The poison sheet, aiming at 6 years life, is 1.3 mm in thickness.
- (2) The poison sheet is asymmetrically inserted 2.5 cm from the viewed surface as shown in Fig. 2.

The Cd poison sheet in the moderator will be mainly irradiated by neutrons, and also influenced by large number of thermal cycles due to cooling (20 K–room temp. in worst case) in operation. Those cycles may cause some kinds of defects in the Cd sheet as in the Cd liner of the cold neutron source of KENS [14]. In order to ensure mechanical, chemical and material stabilities of Cd in a flowing supercritical hydrogen at 20 K, we decided to adopt an Al clad on both sides of the Cd poison sheet. The Cd poison sheet clad in Al needs to be fixed inside the moderator chamber ($12H \times 13W \times 6.3T \text{ cm}^3$) [5,16] made of Al alloy. It is important to obtain a tight bonding between Cd and Al because of the deformation and exfoliation due to the thermal shrinkage and radiation damage, etc. It seems not to be easy to bond these materials due to the low melting point of Cd and the chemically stable Al_2O_3 layer on Al plate.

Following are the descriptions of procedures and results of the experiments by several methods such as explosive welding, heating-up Al vessel containing solder of 70%Cd–30%Zn alloy for Al, cold spraying Al powder on Cd plate, and cold rolling for bonding process as well as HIP processing which was reported in the recent ICANS-XVI meeting [17] for fabrication of

the Cd poison sheet clad with Al. In addition, the method of ultrasonic testing (UT) is studied to establish a non-destructive inspection method to check bonding status of Al and Cd.

2. Experiments and results

2.1. Explosive welding

Fig. 3 shows the principle of an explosive welding method. A plate of an intermediate cladding material is located in parallel with a base plate and at a distance so as to be accelerated enough by explosion toward the base plate. After firing a detonator, explosion waves propagate and force the cladding plate towards the base plate. Both plates strongly collide and generate a metal jet. The metal jet cleans up the surfaces of both plates and exposes a pure metal. As the result, both plates can make a rigid metal bond with each other. Since the metal jet propagation velocity oscillates, the joint interface of both metal that is welded by the explosion method shows a wave form.

In the present work, four kinds of tests were performed using 40 mmW × 90 mmL × 1 mm Cd plates with a different geometrical configuration of Al plate, as shown in Table 1. The main results are as follows:

Case 1: A single pure Al plate was used and located 5 mm distant from a Cd plate on a 5 mm thick steel anvil in order to confirm an explosion welding method. The Cd plate was covered with 3 mm thick steel plate on which the explosive was set. PAVEX with thickness of

Conception of explosive welding

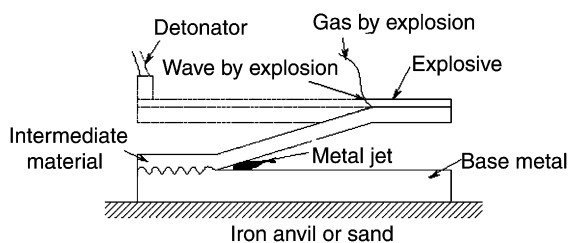


Fig. 3. The principle of explosion welding.

10 mm and weight of 45 g was used on the steel plate. Properties of the PAVEX is shown in Table 2. The detonation was fired at the edge of the steel plate.

An optical microscope was used to check the Al/Cd interface and it was found that the two plates were well bonded by explosion [17].

Case 2: Bonding of both sides of a Cd plate was attempted with two Al plates of 1 mm thickness: one was located at a distance of 5 mm from the Cd plate and the other was beneath the Cd plate on the anvil of a 5 mm thick aluminum and a 18 mm thick iron plate. The upper Al plate was covered by a steel plate on which the PAVEX with thickness of 20 mm and total weight of 124.8 g was used. In order to avoid bonding Al with the plates of the upper steel cover and the lower steel anvil, both interfaces were covered with paper. Detonation was fired at the edge of the steel plate as like Case 1. After explosion, it was found that the Al plates were bended and the lower Al plate had a small crack near the Cd edge at the opposite side of the detonator.

The explosion result was investigated with the ultrasonic test (UT) with the equipment of the Mi-scope of Hitachikenki Fine Tech Co. Ltd. using a 25 MHz probe and a working distance of 15 mm. Then, the sample was treated with thermal-cycles between liquid nitrogen and water, and the sample was cut down into four pieces. There were not found any cracks along the Al/Cd interfaces but found by eyes along the Al/Al interface near the edges. Each section was observed by a color check. The surface of the samples were polished and observed with an optical microscope, SEM and EPMA.

Figs. 4(a)–(c) show the ultrasonic echo patterns reflected from a typical depth to the front side where the PAVEX was located. The dark area of Fig. 4(a) is the outside of the gate because the sample was bent. Fig. 4(b) shows a good bond between Al and Cd at the upper interface except for a local region near the detonator. Fig. 4(c) indicates the defect at the lower interface. These defects are more clear in Fig. 4(e): we can see the defects at the both sides of Cd plate near the tail as well as the head. Figs. 4(d) and (f) reflect the pattern of Fig. 4(e) according to the large working distance. From these results, defects of the interface of Al plate were also observed near the detonator and the tail.

Table 1
Condition of explosion welding test

	To check	Configuration	Al plate	PAVEX thickness
Case 1	Principle of joint	Cd over Al	120 mmW × 200 mmL × 1 mm t	10 mm
Case 2	Two plate bonding	Al over Cd on Al	120 mmW × 200 mmL × 1 mm t	20 mm
Case 3	Detonation position effect	The same as above	120 mmW × 200 mmL × 2 mm t	20 mm
Case 4	Perfect bonding of both sides	Cd plate is separated from both Al plate	120 mmW × 200 mmL × 1 mm t 5 mm t	30 mm

Table 2
Properties of PAVEX

Explosive	Particle velocity (m/s)	Detonation pressure (kbar)	Detonation velocity (m/s)	Density (g/cm ³)	Adiabatic exponent (γ)
PAVEX	741	9.39	2400	0.58	1.98

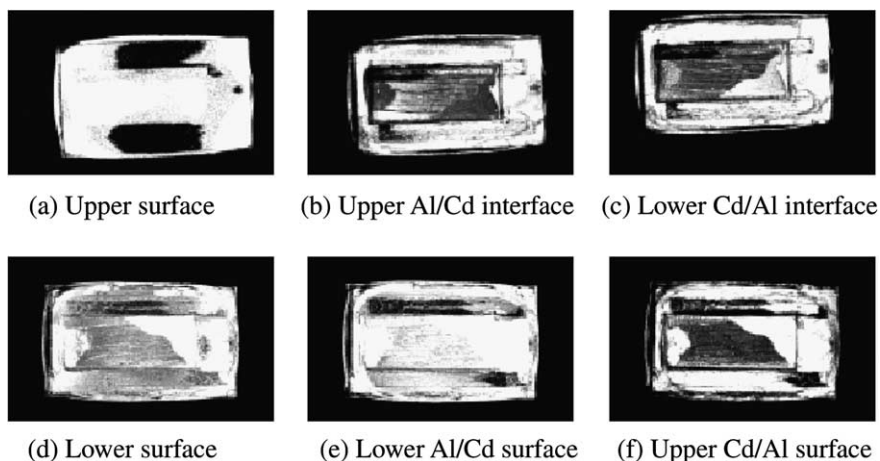


Fig. 4. Ultrasonic diagnostic of Cd/Al plate for Case 2. (a)–(c) are the echo patterns reflected to the front side and (d)–(f) to the behind side.

Fig. 5 shows the picture of the cut and color checked sample. The color check exposed more clearly the defects at the interfaces between the Al plates and only one defect at the Cd/Al interface near the center of the Cd plate.

The optical micrograph of Fig. 6 shows that the major part of the upper Al/Cd interface has no cracks except in the lower region as the UT indicated. The SEM picture of Fig. 7 shows that the upper interface is typically waving as a proof of good bonding by the explosion method. The EPMA showed that thickness



Fig. 5. Picture of cut and color checked sample.

of the Al–Cd mixing (or diffusion) layer at the well-bonded interface as shown in Fig. 7 was about 20 μm and that a part of cadmium shown in Fig. 6(b) was broken near edge by explosion.

Case 3: In order to remove a crack of the Al plate near the tail due to the explosion, Al thickness was increased to 2 mm. Detonation positions were selected at the same position as the plate edge or at the center of the sample.

The explosion experiment resulted in that the crack size on the Al plate was reduced. In this case, we polished the sample surface before UT. Therefore, the echo pattern became clearer than that of Case 2 as shown in Fig. 8. Fig. 8(a) shows a defect at the upper Al/Cd interface near the detonation. That defect seem to be caused by a bonding defect of the Al plates due to too large explosion force near the detonation. Fig. 8(b) shows there is a defect in the wide region at the lower Cd/Al interface. This defect may come from a weaker explosion force. The detonation fired at the center produced large defects not only at the lower Cd/Al interface but also at the upper Al/Cd interface, while a part of the plates far from the detonation was well bonded. These results indicate that the detonator should be located far from Cd plate and thicker Al plates seem to be better to reduce a crack on the Al plate.

Case 4: In this case, we separated both Al plates from the Cd plate by 5 mm in order to obtain a sufficient

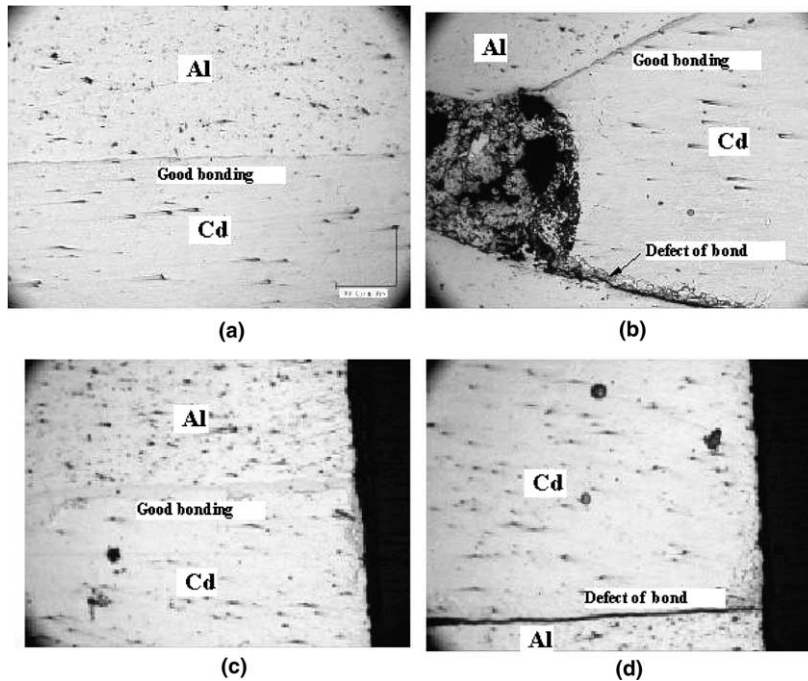


Fig. 6. Optical microscopic picture of Cd/Al plate for Case 2. (a) Optical microscopic picture of cross-section perpendicular to main axis, (b) optical microscopic picture near Cd edge of cross-section along main axis, (c) optical microscopic picture near the center of cross-section along main axis and (d) optical microscopic picture near the center of cross-section along main axis.

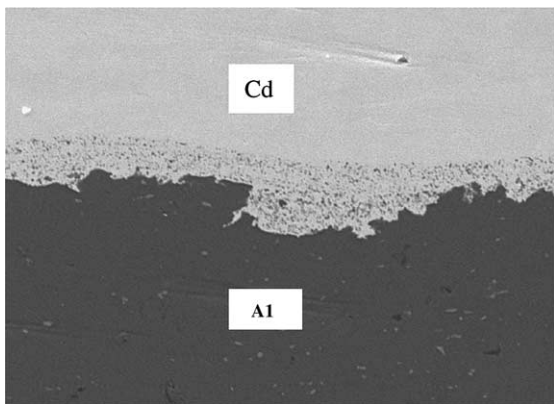


Fig. 7. SEM picture.

bonding strength from the explosion in both interfaces. The upper Al plate was covered by a steel plate on which the PAVEX with thickness of 40 mm and total weight of 480 g was used. The collision pressure was estimated to be 5.88 GPa with the impedance matching method [18] and the flyer plate velocity V_p that was estimated to be 660 m/s according to Chadwick's formula [19]:

$$V_p = V_d \cdot 0.612R / (2 + R),$$

where V_d is the detonation velocity and R is the mass ratio of the PAVEX to the aluminum plate.

Fig. 9 shows the photographs of the sample after explosive welding. There are many ripples of detonation on the surface. These ripples made the UT difficult to observe the defects because they produced multi-scattered echo pattern. Accordingly, we made the UT from the back surface where there are fewer deformations. But, the echo was not clear enough to conclude that there was no defect. Cross-section examination reveals a good welding, although there was a slight bend in the cadmium plate and a motion of Al mass due to the detonation. Good welding was certified by observing the sample with the SEM as shown in Fig. 10 where the interface was characterized by the typical waving pattern of explosion welding.

From these experiments, we concluded that the explosion method is promising to fabricate the Cd poison plate, although deformation of both Al and Cd plates is a problem since it would represent for us a hard additive fabrication task to shape the plates into a flat plate of a thinner thickness, i.e. with a target accuracy 0.5 mm. The next task is to seek optimum conditions to reduce such a deformation.

2.2. Use of Cd solder

It is known that material of 70%wt Cd and 30%wt Zn is a solder for Al. Material properties of this Cd solder

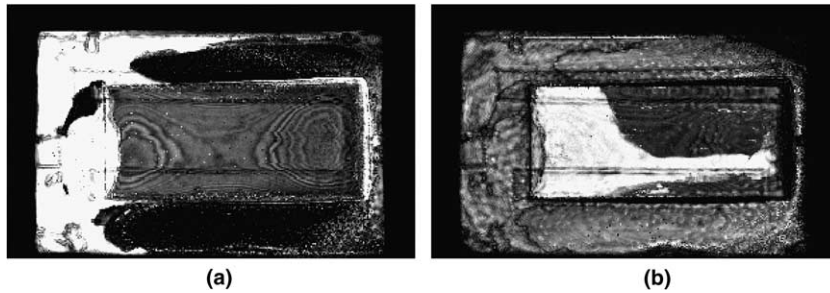


Fig. 8. Ultrasonic diagnostic for Case 3 of explosion welding test. (a) Echo from the upper Al/Cd interface; (b) echo from the lower Cd/Al interface for sample with edge detonation for sample with edge detonation.

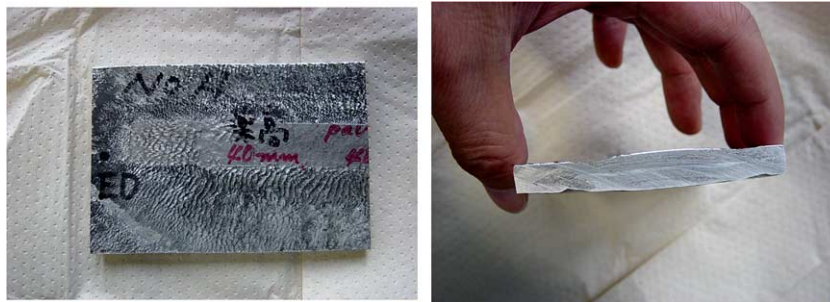


Fig. 9. Cross-section near Cd edge of Case 4 sample of the explosion welding test.

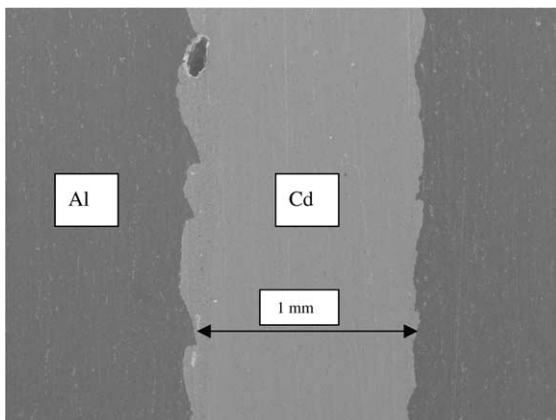


Fig. 10. SEM micrograph of Case 4 sample of the explosion welding test.

are summarized in Table 3. Therefore, it can be used for a poison sheet instead of pure Cd if we can obtain a good bonding between the solder and the Al case. The advantage using this solder is that the manufacturing method is easier than the other ones.

We have tested the method of heating up the Cd solder, the SP-27 made by Nihon Almit Co. Ltd., put in a Al-6061 case covered with a Al-6061 cap of which the inner volume was $100 \times 100 \times 1 \text{ mm}^3$ and thickness was

Table 3

Material property of a Cd solder for Al

Chemical ratio	Cd: 70wt% Zn: 30wt%
Solidus	About 266 °C
Liquidus	About 292 °C

5 mm. The pretreatment to deoxidize inner surfaces of the Al case and the cap was made with a corrosive solution (20 ml distilled water, 20 ml HCl (32%), 20 ml HNO₃ (65%) and 5 ml HF (40%)), instead of using a normal flux. The Al case and cap were not enclosed with any bonding methods in order to give the case a flexibility to accommodate volume expansion of the solder. The samples were heated up to about 300 °C (Case 1) and 340 °C (Case 2) using a vacuum chamber in which the pressure was about 10^{-5} Torr.

In Case 1, the bonding was apparently good. However, thermal cycles between liquid nitrogen temperature and water easily separated the Al–Cd bond. In Case 2, though the bonding was also apparently good, the Cd solder leaked to the outside of the Al case. Moreover vaporization of the Cd solder was found in an inner surface of the vacuum chamber. Fig. 11 shows a ultrasonic echo pattern for the Case 2 sample indicating bonding between the solder and the Al case. The central region seems not to exhibit such good bonding. Fig. 12 shows

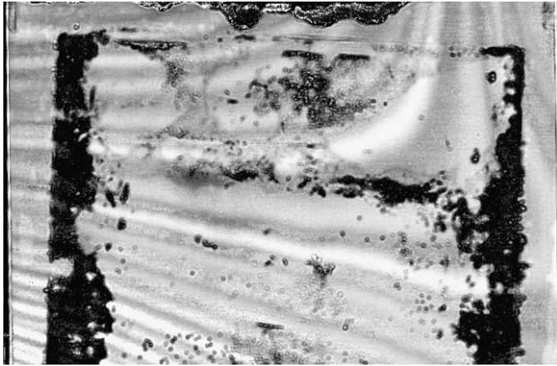


Fig. 11. UT echo pattern from the interface between 70% Zn and 30% Zn solder and Al clad of the Case 2 sample.

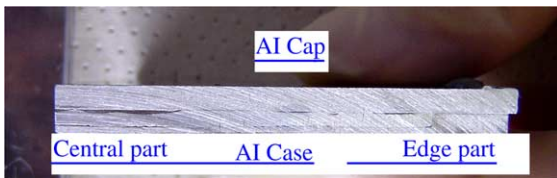


Fig. 12. Cut cross-section of the Case 2 sample.

a cutting view of the Case 2 sample. A few un-bonded parts (likely due to shrinkage cavities) were found in the cap side between the solder and the Al case, though it shows better bonding in other parts. The cavities may be caused by inadequate solder volume and weight pressing the cap from above.

From these experiments, it is found that there are such issues to be solved as casing manufactures, suppression of any cavities, better solder wettability to extend on the whole surface and so on. However, this method would be one of the good bonding methods.

2.3. Cold spraying

Cold spraying is one of the candidate methods to bond Cd and Al in order to avoid the severe problems of evaporation of poisonous Cd. In the SNS project [20], Cd powders are sprayed on an Al surface in order to make a de-coupler. But such method would hardly be accepted by any Japanese factory, fearing a toxic problem of Cd vapors. In reverse, we adopted the method to cold-spraying Al powders on a Cd plate by using nitrogen gas as a powder-spraying power. Gas temperature was controlled by a heater. Atomized Al powders of which the average particle diameter was 13 μm were used in the experiments. Cold spraying condition was surveyed with small samples of Cd (99%) and Zn by

changing a nitrogen-gas pressure to spray powder between 2.5 and 3.5 MPa and a nozzle temperature between 250 $^{\circ}\text{C}$ and 350 $^{\circ}\text{C}$. The Cd temperature was measured to certify that remains below the melting temperature of 320 $^{\circ}\text{C}$.

Since one coat of sprayings made only a 0.05–0.3 mm thick layer of Al on the Cd plate, multiple Al-powder sprayings should be made to form a Al layer of about 1 mm thickness. It was also found that the optimum condition for the cold-spray was 3 MPa and 300 $^{\circ}\text{C}$. Then, a test for the real-size sample of Cd plate of $100 \times 100 \times 1 \text{ mm}^3$ was made to confirm the method. The layer was observed with the optical microscope. It indicated that accumulated layer was porous and this tendency was more prominent near the outside of the Al surface than near Al/Cd interface.

2.4. Cold Rolling-Clad

The Cold Rolling-Clad technique is frequently used for Al goods. This method makes fresh surfaces on the both plates of Al and a counter material: fresh material-surfaces are chemically active and produce good bonding Al on the counter material. We have tested this method by using a 56 mmL \times 110 mmW \times 3.5 mmT Cd plate located in the central vacant place of the Al vessel space with 199 mmL \times 128 mmW \times 8.5 mmT in the outer dimension. The Al vessel was covered by 2.5 mmT Al plate. The lid plate and the lower vessel was bonded by electron beam welding in a vacuum. Rolling was made in the axial direction. Tests were made for six kinds of samples by changing number of passes from 1 to 13 and a rolling temperature. Maximum rolling rate was 26%. However, UT showed that bonding state is doubtful in all cases. The present method may have resulted in failure because no pretreatment was used for removing the Al_2O_3 layer on the Al vessel. It indicates that pretreatment is required to lead to successful cold rolling clad.

3. Discussion and concluding remarks

Cd poison plate is the most convenient to obtain sharp neutron pulses because the cut-off energy is very high compared to Gd. However, the Cd plate should be covered by Al clad. Both metals should be satisfactorily bonded to remove nuclear heat of the poison plate in the hydrogen moderator. In the present work, we tested several bonding techniques such as explosive, heating up of a solder of 70%Cd–30%Zn in the Al case, cold spraying, cold rolling. In the previous work [17], the HIP method was examined.

We found that bonding was obtained in case of the explosive welding and heating up a solder of 70%Cd–30%Zn in Al case although it was not ideally good.

The HIP method with 300 °C, 100 MPa and 1 h processing time gave a bond in the case of using an intermediate thin layer of Zn or Mg together with the pretreatment of chemical etching against the Al surface. These results indicate that the Al₂O₃ layer of the Al plate plays an important role in bonding with the Cd plate at low temperature. In the explosion welding method, the explosion gas jet blows away the Al₂O₃ layer and cleans up both surfaces of the Cd and Al plates. In the soldering method with 70%Cd–30%Zn in the Al case and the HIP method, chemical etching is effective to activate the Al surface. In the cold spraying method, the kinetic energy of Al powder which was given by nitrogen gas produced sufficient mechanical-bonding power. However, this method has a fatal problem accompanied with a porosity. In the cold rolling clad method, with no pretreatment of cleaning up the Al surface fails in the bonding. At the present time, it is concluded the methods of explosion welding and soldering are the most promising for fabricating the Cd poison sheet. The improved HIP method with pretreatment of Al surface is the next candidate.

As for a non-destructive defect inspection, UT is very powerful. Moreover, it showed the defects in wider regions than the SEM and the optical microscope indicated. But, it is necessary to improve the method in cases of thicker samples with some deformation. In order to reform the explosion welded plate with some ripple, we have to measure the deformation of the plate. The further study of the reliability of the UT measurements in the vertical direction will provide means to improve the fabrication techniques of the poison sheet.

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