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Evaluation of interface strength between metal and ceramics to be utilized for development of fusion reactor components

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A B S T R A C T

Yttrium oxide coating was successfully made on V–4Cr–4Ti–Y alloy using DC-sputter coating followed by heat treatment at 1000 °C. Adhesive shear strength of the yttrium oxide coating was evaluated by means of scratch tests. The Weibull analysis of the shear strength of the interface between the coating and alloy made by DC-sputter coating indicated that surface polishing affects the evaluation of the strength. In the case of the same surface roughness about 0.1 μm, it was noted that small additions of yttrium to V–4Cr–4Ti would improve adhesive property of the interface between yttrium oxide coating and vanadium alloys. Yttrium oxide formed at the very beginning of the coating process could result in better adhesive properties.

1. Introduction

Coating and bonding techniques between different materials are essential to the field of technology including fusion reactor engineering. Protective coating layer on the structural materials would improve performance of components in corrosive environment. The interfaces between different materials, typically metals and ceramics, are of interest for various fields of engineering. Fundamental understanding of metal and ceramics interfaces would provide us with a useful guideline to develop the advanced coating and bonding technique for fusion reactor components. Since self-cooled blanket design with liquid lithium is primarily considered when vanadium alloys are used as structural materials in fusion reactors, it is vital to reduce the pressure drop. Insulator coating inside of the lithium channel is a major option for solving this issue. Early assessments have made Y_2O_3 and Er_2O_3 prime candidates [1]. Selection of coating methods that can be applied to the complex structures like tubing channels and elbows are now under consideration. A good knowledge of the mechanical properties of the interface between the ceramics and vanadium base alloy is essential for reliable development of the coating including their preparation methods. In this paper, yttrium oxide is used for fundamental study of the metal ceramics bonds in terms of strength.

2. Experimental procedure

2.1. Materials and preparation of coating layers

Several methods of the coating and bonding were attempted to make the interface between vanadium alloys and yttrium oxide, these included low-pressure plasma spraying, solid-sate diffusion bonding, dipping using metal organic decomposition, and physical vapor deposition like sputter coating. In this paper, results of the coating using DC sputter coating are described. The materials used as substrate were V–4Cr–4Ti and V–4Cr–4Ti–0.2Y alloys. Chemical compositions of the alloys are shown in Table 1. Fabrication procedures of the alloys were described in references [2,3]. The platelets of the alloys with 10 mm width and 20 mm length with 2 mm thick were annealed at 950 °C for 1 h in a vacuum to obtain recrystallized condition for the sputter coating. The surface was polished on a series of diamond slurry. Electropolishing using a solution of 20%-H_2SO_4 and 80%-CH_3OH at 20 °C was carried out before the coating. The DC-sputter coating with 12 W power was carried out using yttrium metal as target (50 mm in diameter) in argon gas atmosphere about 7 Pa. Heat treatment after the sputter coating was carried out in a vacuum at elevated temperature up to 1000 °C to improve crystallinity of the coating.

2.2. Evaluation of the coating layers

A grazing incidence X-ray diffraction with a rotating specimen stage (JEOL, JDX3530) was used to characterize the crystallographic structure of the coating. The incident angle was set to 1° and Cu Kα X-ray tube was used at 40 kV and 40 mA. Thickness of the coating was measured by a surface profiler (KLA-Tencor,

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Table 1
Chemical analysis of vanadium alloys in weight percentage (O, N, C in wt. ppm).

Alloy	Cr	Ti	Si	Al	Y	O	N	C	V
V–4Cr–4Ti	4.03	3.73	–	–	–	114	122	50	Bal.
V–4Cr–4Ti–02Y	4.32	4.35	0.01	<0.002	0.24	47	85	70	Bal.

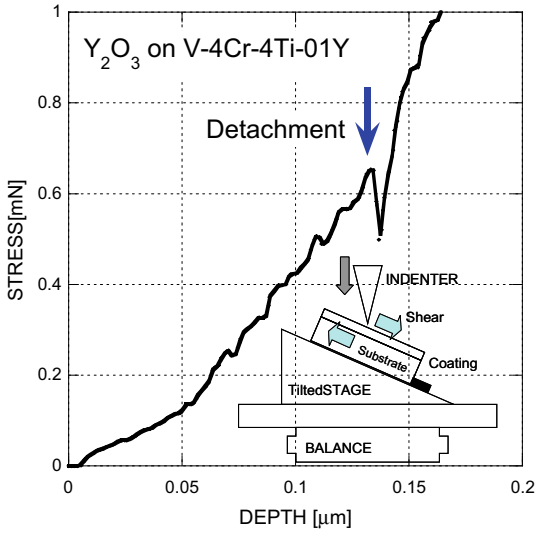

Fig. 1. Typical depth and load profile of scratch test for measuring of interface adhesive shear strength.

P-10) followed by Cs-ion sputtering using a secondary ion mass spectroscopy (SIMS, ULVAC-PHI, PHI-6600L). The sputtered ion was analyzed by a quadrupole mass analyzer. The sputtering by Cs-ion was continued until yttrium signal was depressed. The depth profile indicated the thickness of the coating was about 0.5 μm.

2.3. Evaluation of interface strength

Shear strength of the interface was measured by scratch test following the Japanese Industrial Standard JIS-R3255 'Test Methods for Adhesion of Thin Films on Glass Substrate'. A ruby indenter was forced into the specimen placed on tilted stage using thin film material evaluation system, MH4000 (NEC San-ei Instruments, Ltd.). The maximum adhesive shear stress τ was calculated using the following equation:

$$\tau = \frac{((1-2\nu)\cos\theta - 3\pi(\frac{1}{2}+\frac{\nu}{8})\sin\theta)W}{2\pi r(2r\delta-\delta^2)^{1/2}}, \quad (1)$$

where ν is Poisson's ratio, δ is depth of the indentation, W is critical load just before immediate drop of the load as shown in Fig. 1, θ is 30°, tilt angle of the stage, r is 5 μm, radius of the ruby tip.

3. Results and discussion

3.1. Characterization of coating layers

Fig. 2 shows XRD spectrum of the coating made by the DC-sputter before and after heat treatment. The diffraction peak around 29.15° is corresponding to (222) plane of Y_2O_3 crystal [4]. The full-width of the half-maximum (FWHM) is smaller at higher temperature of heat treatment. The FWHM of (222) peak after heat treatment at 600 °C and that at 1000 °C were 0.76° and 0.54°, respectively. The sharper peak indicated that the crystallinity of the coating was better after heat treatment at 1000 °C. Peak shift corresponding to compressive strain or smaller d-spacing of the

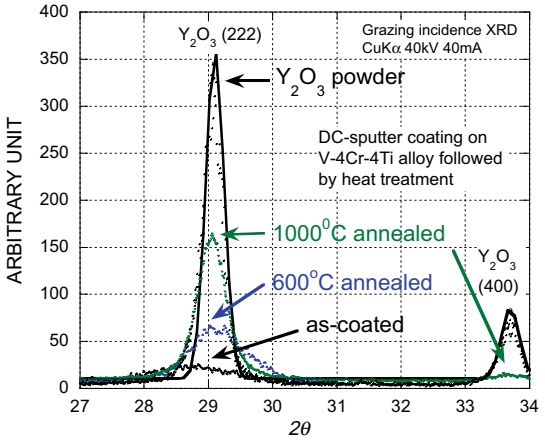

Fig. 2. X-ray diffraction patterns of yttria coating on V–4Cr–4Ti alloy followed by heat treatment at 600 or 1000 °C for 1 h. XRD pattern from Y_2O_3 powder and in the as-coated condition is shown for comparison.

(222) plane compared to as-coated condition is observed. Thermal liner expansion of the vanadium was larger than that of Y_2O_3 [5,6], so that the residual thermal stress should be compression along to the interface plane. It might be also possible that the stoichiometry of the oxide layer affects the peak shift. The d-spacing of the corresponding peaks of $YO_{1.335}$ and $YO_{1.458}$ were reported to be at 0.335 and 0.314 nm, respectively [7]. The d-spacing observed 0.310 nm for as-coated and 0.308 nm for coating followed by 600 °C annealing are corresponding to $YO_{1.477}$ and $YO_{1.491}$, respectively, when the d-spacing represented the stoichiometry of the coating. The observed peak shift indicates that the heat treatment made the yttrium oxide of the coating closer to the stoichiometry of Y_2O_3.

3.2. Strength of the interface

As shown in typical stress and displacement depth curve in Fig. 1, the immediate drop of the stress is observed as indicated arrow should correspond to detachment of the interface due to the shear stress by the indentation. The average adhesive shear strength of the yttrium oxide coating on vanadium alloys were varied from 54 to 83 MPa depending on the alloy composition and surface roughness. The average roughness (Ra) of the surface in the as rolled and mechanically polished condition, of the alloys were 0.08–0.22 μm and 0.04–0.07 μm, respectively.

Fig. 3 shows the adhesive shear strength of yttrium oxide coating on vanadium alloys using Weibull plot. The shape parameter, m is defined as the slope, S is survival probability of failure. Surface roughness affected the actual shear stress applied to the interface. As defined in Eq. (1), θ is fixed to 30°, but the value may vary from 25° to 35° in typical case of Ra = 0.2 μm because of their roughness. The corresponding shear stress that have the roughness Ra = 0.2 μm varied about 20%. Brittle material like the ceramics coating breaks at the weakest part, so that the shear strength of the relatively rough specimen showed lower than that of smooth specimen as shown in Fig. 3. Comparing same surface condition (Ra = 0.07 or 0.08 μm), the coating on V–4Cr–4Ti–02Y alloy shows larger values of m and average shear strength than that on V–4Cr–4Ti alloy. This indicates that small additions of yttrium affect the adhesion properties of the coating. It may be related to oxidation behavior of V–4Cr–4Ti–Y alloy. The oxide film on V–4Cr–4Ti–02Y alloy formed at elevated temperatures in flowing helium gas was dense and hard compared to V–4Cr–4Ti alloy without yttrium additions [8,9]. Sputter yield from the coating during SIMS analysis indicated that the mixture of yttrium oxide and vanadium oxide were formed at the very beginning of the coating may result in better adhesive properties.

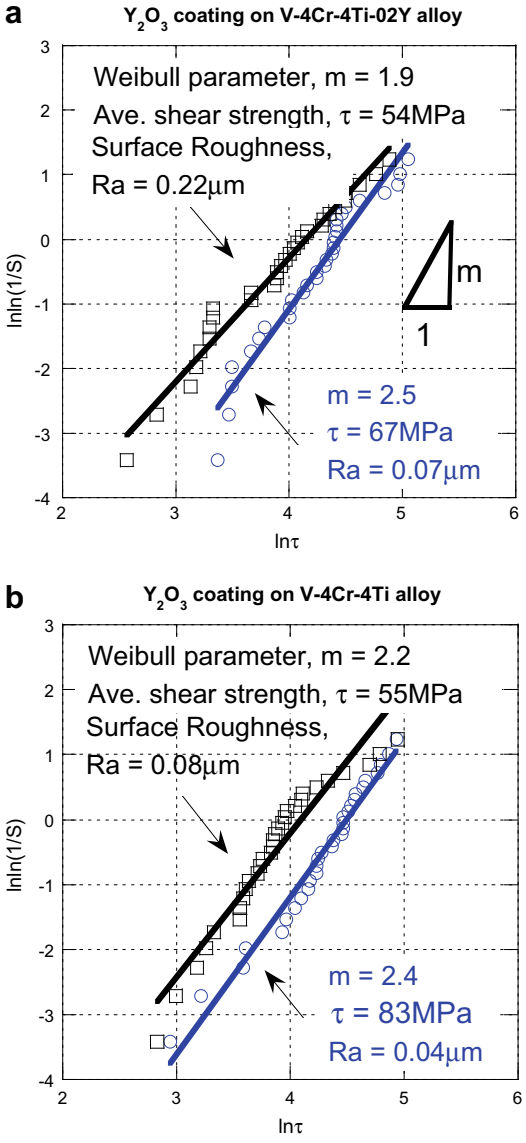

Fig. 3. Shear strength versus survival probability of Y_2O_3 coating on V–4Cr–4Ti–02Y alloy (a), and V–4Cr–4Ti alloy (b) on Weibull plot. (ln is a natural logarithm.)

4. Summary

Yttrium oxide coating was successfully made on vanadium alloys by means of DC-sputter coating. To evaluate reliability of the coating, adhesive property was measured by scratch test. The Weibull analysis of the shear strength of the interface between yttrium oxide coating and vanadium alloys made by DC-sputter coating indicated that surface polishing affects the strength evaluation. In the case of the same surface roughness about 0.1 μm, it was noted that small additions of yttrium to V–4Cr–4Ti would improve adhesive property of the interface between yttrium oxide coating and vanadium alloys. Evaluation method and improvement in reliability of the coating are still needed for engineering application of the material system, such as insulators coating of vanadium alloys.

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References

[1] B.A. Pint, P.F. Tortorelli, A. Jankowski, J. Hayes, T. Muroga, A. Suzuki, O.I. Yeliseyeva, V.M. Chernov, J. Nucl. Mater. 329–333 (2004) 119.
[2] T. Nagasaka, T. Muroga, M. Imamura, S. Tomiyama, M. Sakata, Fus. Technol. 39 (2001) 659.
[3] T. Chuto, M. Satou, A. Hasegawa, K. Abe, T. Nagasaka, T. Muroga, J. Nucl. Mater. 307–311 (2002) 555.
[4] JPDS, powder diffraction file, inorganic substances, Pennsylvania: JPDS International Center for Diffraction Data, 1992.
[5] Y.S. Touloukian, R.K. Kirby, R.E. Taylor, P.D. Desai, Thermal Expansion: Metallic Elements and Alloys, IFI/Plenum, New York, 1975.
[6] Y.S. Touloukian, R.K. Kirby, R.E. Taylor, T.Y.R. Lee, Thermal Expansion: Nonmetallic Solids, IFI/Plenum, New York, 1975.
[7] A. Solov'eva, Inorg. Mater. (Engl. Transl.) 21 (1985) 701.
[8] M. Fujiwara, M. Satou, A. Hasegawa, K. Abe, J. Nucl. Mater. 283–287 (2000) 1311.
[9] M. Satou, T. Nagasaka, T. Hino, M. Fujiwara, T. Muroga, T. Iikubo, K. Abe, 21th IAEA Fusion Energy Conference, FT/P5-34, ISBN 92-0-100907-0 (2006).