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TRIAL FABRICATION AND PRELIMINARY  
CHARACTERIZATION OF ELECTRICAL  
INSULATOR FOR LIQUID METAL SYSTEM

March 1995

Masaru NAKAMICHI, Hiroshi KAWAMURA  
and Rokuro OYAMADA

日本原子力研究所  
Japan Atomic Energy Research Institute

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Trial Fabrication and Preliminary Characterization of  
Electrical Insulator for Liquid Metal System

Masaru NAKAMICHI, Hiroshi KAWAMURA and Rokuro OYAMADA

Department of JMTR Project  
Oarai Research Establishment  
Japan Atomic Energy Research Institute  
Oarai-machi, Higashiibaraki-gun, Ibaraki-ken

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In the design of the liquid metal blanket, MHD pressure drop is one of critical issues. Ceramic coating on the surface of structural material is considered as an electrical insulator to reduce the MHD pressure drop. Ceramic coating such as  $Y_2O_3$  is a promising electrical insulator due to its high electrical resistivity and good compatibility with liquid lithium.

This report describes the trial fabrication and preliminary characterization of electrical insulator for a design study of the liquid metal system. From the results of trial fabrication and preliminary characterization, it is concluded that densified atmospheric plasma spray  $Y_2O_3$  coating with 410SS undercoating between 316SS substrate and  $Y_2O_3$  coating is suitable for  $Y_2O_3$  coating fabrication.

Keywords: Liquid Metal Blanket, MHD Pressure Drop, Ceramic Coating,  $Y_2O_3$ , Atmospheric Plasma Spray, Densification Treatment, Electrical Insulator, Chemical Densified Coating, Undercoating, 410SS

液体金属ブランケットにおける電気絶縁材としての  
セラミックコーティング膜の試作及び予備特性評価

日本原子力研究所大洗研究所材料試験炉部

中道 勝・河村 弘・小山田六郎

(1995年1月31日受理)

液体金属ブランケット設計では、MHD圧力損失が大きな問題となっている。その解決策として、構造体への電気絶縁材としてセラミックコーティング膜の施工が考えられている。セラミックコーティング膜としては、高電気絶縁性及びリチウムとの優れた共存性の観点から $Y_2O_3$ が候補として挙げられている。

本報告書は、電気絶縁材としてのセラミックコーティング膜の試作及び予備特性評価についてまとめたものである。本試作及び予備特性評価の結果から、母材SUS316上にSUS410をアンダーコーティングし、その上に緻密処理した $Y_2O_3$ の大気プラズマ溶射膜を施工することにより、耐熱衝撃性に優れた $Y_2O_3$ 膜を施工できることが明らかになった。

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## 1. Introduction

In the design of the liquid metal blanket, Magneto-Hydro-Dynamic (MHD) pressure drop is one of critical issues. Ceramic coating on the surface of structural material is being considered for an electrical insulator to reduce the MHD pressure drop. Ceramic coating such as  $Y_2O_3$  is a promising electrical insulator due to its high electrical resistivity [1] (see Fig.1) and good compatibility with liquid lithium [2] (see Fig.2). The purposes of this study are to develop ceramic coating method and to obtain material database of the coating.

The following four methods have been tried for fabrication of  $Y_2O_3$  coating on 316SS substrate. Technical flow of trial fabrication of  $Y_2O_3$  coating on 316SS substrate is shown in Fig.3.

case 1 : Chemical Densified Coating (CDC)

case 2 : Atmospheric plasma spray

case 3 : Atmospheric plasma spray with densification treatment

case 4 : Atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment

The CDC [3] method is a promising method for fabrication of ceramic coating because of its capability to form densified and firm coating either on outer or inner surface of a tube [4-6]. Furthermore, this method is able to apply various materials as a coating and to coat on the surface of metallic materials.

In various industries, ceramic and metal coating by the atmospheric plasma spray are useful for heat resisting coating and anti corrosion coating, and this method is able to apply various materials as a coating and to coat thick coating.

In case 3, the coating by the atmospheric plasma spray was densified by impregnation with  $Y(NO_3)_3$  solution and fired at 500°C for enclosure of pore in atmospheric plasma spray  $Y_2O_3$  coating.

In case 4, 410SS was coated on 316SS substrate as an undercoating between the substrate and  $Y_2O_3$  coating to reduce crack and peeling of  $Y_2O_3$  coating.

## 2. Trial Fabrication and Preliminary Characterization

### 2.1 Chemical Densified Coating

#### 2.1.1 Trial Fabrication Method

The fabrication process in case 1 was shown in Fig.4. In the first step, the surface of 316SS substrate was degreased by methyl alcohol and  $Al_2O_3$  grit blasted to improve the adhesion between 316SS substrate and  $Y_2O_3$  coating. 316SS (\*50x130x4mm) as substrate was made by NIIGATA STAINLESS CORPORATION (see Table 1). In the second step, ceramic slurry was applied on

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316SS substrate. Ceramic slurry was the solution of  $Y_2O_3$  particles (average particle size :  $2.53\mu m$ ) and  $Y(NO_3)_3$  solution and  $H_2O$  in the ratio of 1:1:1 (weight).  $Y_2O_3$  particles for ceramic slurry was made by SANTOKU METAL INDUSTRY Co. Ltd. (see Table 2).  $Y(NO_3)_3$  solution was made by DAIICHI KIGENSO KAGAKU KOGYO Co. Ltd. (see Table 3). In the third step, 316SS substrate with ceramic slurry was heated for one hour at  $500^\circ C$  under atmospheric condition in an electric furnace. The rate of heating and cooling were  $500^\circ C/h$  and  $100^\circ C/h$ , respectively. In the fourth step,  $Y_2O_3$  coating was densified by impregnation with  $Y(NO_3)_3$  solution and heated one hour at  $500^\circ C$  in order to close open pores, because  $Y_2O_3$  coating was porous at the end of the third step. The rate of heating and cooling were the same as the third step. Furthermore, densification treatment such as the fourth step was repeated 15 times.

### 2.1.2 Preliminary Characterization

#### 1) Thickness of Coating

The thickness of the  $Y_2O_3$  coating was about  $23\mu m$ . Ceramic slurry solidified at about 10 minutes after mixture of  $Y(NO_3)_3$  solution and  $Y_2O_3$  particles. Therefore, in this method, it was difficult to control the thickness of coating. Coating thickness attained was only about  $23\mu m$ .

#### 2) Metallographical Observation

The photographs of a cross section in the  $Y_2O_3$  coating by optical microscope and scanning electron microscope (SEM) were shown in Fig.5 and 6, respectively. From these metallographical observations, many cracks through the 316SS substrate surface were observed in the  $Y_2O_3$  coating.

## 2.2 Atmospheric Plasma Spray

### 2.2.1 Trial Fabrication Method

This fabrication process in case 2 was shown in Fig.7. In the first step, the surface of 316SS substrate was degreased by methyl alcohol and  $Al_2O_3$  grit blasted to improve the adhesion between 316SS substrate and  $Y_2O_3$  coating. In the second step,  $Y_2O_3$  was coated by atmospheric plasma spray.  $Y_2O_3$  particle size was from  $10\mu m$  to  $45\mu m$ .  $Y_2O_3$  particles for atmospheric plasma spray was made by TOCALO Co. Ltd. (see Table 4). Atmospheric plasma spray condition for the  $Y_2O_3$  coating was shown in Table 5. Plasma gas were Ar and  $H_2$ , each flow rate were  $7.0 \times 10^{-4}$  and  $2.0 \times 10^{-4} m^3/s$ , respectively. Plasma current, plasma voltage and spray distance were 550A, 74V and 120mm, respectively. A-3000S made by PLASMA-TECHNIK AG was used as spraying apparatus.

### 2.2.2 Preliminary Characterization

#### 1) Thickness of Coating

The thickness of the  $Y_2O_3$  coating was about  $60\mu m$  with easy control of thickness of  $Y_2O_3$  coating.

## 2) Metallographical Observation

The photographs of a cross section in the  $Y_2O_3$  coating by optical microscope and SEM were shown in Fig.8 and 9, respectively. From these metallographical observations, many pores existed in the  $Y_2O_3$  coating.

## 3) Hardness Test

The results of the micro Vickers hardness of a cross section of the  $Y_2O_3$  coating with load of 0.245N were shown in Table 6. The average of the micro Vickers hardness was 389Hv. MVK-E made by AKASHI SEISAKUSHO Ltd. was used as hardness test.

## 4) Adhesion Test

Specimens for adhesion test and the outline of adhesion test were shown in Fig.10 and 11, respectively.  $Y_2O_3$  was coated about 60 $\mu$ m thickness by atmospheric plasma spray on  $\varnothing$ 25mm cylindrical specimen of 316SS. Firstly, an adhesion specimen with  $Y_2O_3$  coating and a specimen without  $Y_2O_3$  coating were joined by the bonding agent, MASTER BOND EP15 made by MASTER BOND Inc., and heated for two hours at 170°C under atmospheric condition in an electric furnace. Next, the joined specimen was placed in the tensile machine. Adhesion was measured at 2.3mm/min tensile speed in room temperature with three testing specimens. In this test, 18219 made by TESTER-SANGYO Co. Ltd. and T3B1-5T made by MINEBEA Co. Ltd. were used as tensile machine and load cell, respectively.

The results of adhesion test and photograph of appearance after adhesion test were shown in Table 7 and Fig.12. The average of the adhesion strength of the  $Y_2O_3$  coating was 40.4MPa.

## 5) Thermal Shock Test

Thermal shock tests were performed by water quenching from 500, 600, 700 and 800°C. The holding time at each temperature was 30 minutes. The number of testing specimens was two for each temperature.

The photographs of appearance after thermal shock test at 500, 600, 700 and 800°C were shown in Fig.13, 14, 15 and 16, respectively. The photographs of a cross section in the  $Y_2O_3$  coating by optical microscope after thermal shock test at 500, 600, 700 and 800°C were shown in Fig.17, 18, 19 and 20, respectively. The photographs of a cross section in the  $Y_2O_3$  coating by SEM after thermal shock test at 500, 600, 700 and 800°C were shown in Fig.21, 22, 23 and 24, respectively. The results of the thermal shock test was shown in Fig.25.

From the results of the thermal shock test, it was clear that the  $Y_2O_3$  coating was inferior in thermal shock resistivity. In thermal shock test at 500°C, peeling of the coating occurred after one cycle, and peeling of about 50% of the  $Y_2O_3$  coating was observed after 11 cycles (see Fig.13). In thermal shock test at 600 and 700°C, peeling of the all or almost all the  $Y_2O_3$  coating was observed after 2 cycles (see Fig.14 and 15). In thermal shock test at 800°C, peeling of all the  $Y_2O_3$  coating was observed after one cycle (see Fig.16).

## 6) Measurement of Electrical Resistivity

Electrical resistivity of the  $Y_2O_3$  coating was measured from room temperature to 900°C in Ar gas. Specimen and outline for electrical resistivity measurement was shown in Fig.26. Ag paste was applied on the  $Y_2O_3$  coating, and heated for 20 minutes at 150°C. P383 made by TOKURIKI

CHEMICAL Ltd. was used as Ag paste. TOA ELECTRIC Ltd. was used as high resistance meter. The impressed voltage was DC 500V. The results of electrical resistivity measurement were shown in Table 8 and Fig.27. Electrical resistivity of the  $Y_2O_3$  coating is  $1.0 \times 10^{11}$ ,  $5.0 \times 10^9$ ,  $1.6 \times 10^8$  and  $1.0 \times 10^7 \Omega \cdot \text{cm}$  at 200, 300, 500 and 800°C, respectively, which satisfy ITER design guideline ( $>1.0 \times 10^5 \Omega \cdot \text{cm}$ , from 200°C to 800°C).

## 2.3 Atmospheric Plasma Spray with Densification Treatment

### 2.3.1 Trial Fabrication Method

The fabrication process in case 3 was shown in Fig.28. In this fabrication process, atmospheric plasma sprayed  $Y_2O_3$  coating of case 2 was densified by impregnation with  $Y(NO_3)_3$  solution and heated at 500°C for enclosure of pore in the  $Y_2O_3$  coating. The rate of heating and cooling were 500°C/h and 100°C/h, respectively, with repeated densification treatment.

### 2.3.2 Preliminary Characterization

#### 1) Appearance and Metallographical Observation

The photographs of appearance and SEM observation after three times treatment of densification were shown in Fig.29 and 30, respectively. The broken part of specimen in Fig.29 was used metallographical observation. Peeling of about 50% of the  $Y_2O_3$  coating occurred after three times of densification (see Fig.29). In this method, it was unable to fabricate atmospheric plasma sprayed  $Y_2O_3$  coating with densification treatment.

## 2.4 Atmospheric Plasma Spray Undercoated by 410SS between 316SS Substrate and $Y_2O_3$ Coating with Densification Treatment

### 2.4.1 Trial Fabrication Method

This fabrication process in case 4 was shown in Fig.31. In this fabrication process, 410SS was coated by atmospheric plasma spray on the 316SS substrate as an undercoating between the substrate and the sprayed  $Y_2O_3$  coating to prevent the crack and peeling of the  $Y_2O_3$ . 410SS was selected because thermal expansion coefficient of 410SS was close to that of  $Y_2O_3$  (see Fig.32). In the first step, the surface of the 316SS substrate was degreased by methyl alcohol and  $Al_2O_3$  grit blasted to improve the adhesion between the substrate and 410SS undercoating. In the second step, 410SS was applied about 150 $\mu\text{m}$  thick by atmospheric plasma spray. 410SS particle size was from 10 $\mu\text{m}$  to 74 $\mu\text{m}$ . 410SS particles for undercoating of atmospheric plasma spray was made by SHOWA DENKO K.K (see Table 9). Atmospheric plasma spray condition for 410SS undercoating was shown in Table 10. Plasma gas were Ar and  $H_2$ , each flow rate were  $9.2 \times 10^{-4}$  and  $1.5 \times 10^{-4} \text{ m}^3/\text{s}$ , respectively. Plasma current, plasma voltage and spray distance were 550A, 72V and 130mm, respectively. A-3000S made by PLASMA-TECHNIK AG was used as spraying apparatus. In the third step,  $Y_2O_3$  was coated by atmospheric plasma spray with the

same conditions as those in case 2. In the fourth step,  $Y_2O_3$  coating was densified by impregnation with  $Y(NO_3)_3$  solution and heated for one hour at  $500^\circ C$  for enclosure of pore, because  $Y_2O_3$  coating was porous at the end of the third step. The rate of heating and cooling were  $500^\circ C/h$  and  $100^\circ C/h$ , respectively, with 14 times densification treatment.

#### 2.4.2 Preliminary Characterization

##### 1) Thickness of Coating

The thickness of the  $Y_2O_3$  coating was about  $60\mu m$ .

##### 2) Metallographical Observation

The photographs of a cross section in the  $Y_2O_3$  coating by optical microscope and SEM were shown in Fig.33 and 34, respectively. From these metallographical observations, many cracks along the interface between 410SS undercoating and  $Y_2O_3$  coating were existed in the  $Y_2O_3$  coating (see Fig.33 and 34).

##### 3) Hardness Test

The results of the micro Vickers hardness of a cross section of the  $Y_2O_3$  coating with load of  $0.245N$  were shown in Table 11. The average of the micro Vickers hardness was  $518Hv$ . MVK-E made by AKASHI SEISAKUSHO Ltd. was used as hardness test. The hardness was increased from  $389Hv$  to  $518Hv$  by densification treatment in comparison with no-densification treatment  $Y_2O_3$  coating of case 2.

##### 4) Thermal Shock Test

Thermal shock tests were performed by water quenching from  $500, 600, 700$  and  $800^\circ C$ . The holding time at each temperature was 30 minutes. The number of testing specimens was two for each temperature.

The photographs of the appearance after thermal shock test at  $500, 600, 700$  and  $800^\circ C$  were shown in Fig.35, 36, 37 and 38, respectively. The photographs of a cross section in the  $Y_2O_3$  coating by optical microscope after thermal shock test at  $500, 600, 700$  and  $800^\circ C$  were shown in Fig.39, 40, 41 and 42, respectively. The photographs of a cross section in the  $Y_2O_3$  coating by SEM after thermal shock test at  $500, 600, 700$  and  $800^\circ C$  were shown in Fig.43, 44, 45 and 46, respectively. The results of thermal shock test was shown in Fig.47.

In thermal shock test at  $500^\circ C$ , peeling of very small area the  $Y_2O_3$  coating of one specimen occurred after 15 cycles, but this peeling area of the coating was not increased after 30 cycles (see Fig.35). The  $Y_2O_3$  coating of other specimen could stand 30 cycles of the thermal shock test at  $500^\circ C$ . In thermal shock test at  $600, 700$  and  $800^\circ C$ , peeling of almost the  $Y_2O_3$  coating was observed after one cycle (see Fig.36, 37 and 38). From the metallographical observation after thermal shock test at  $500^\circ C$ , many cracks along the interface between 410SS undercoating and  $Y_2O_3$  coating were observed in the  $Y_2O_3$  coating (see Fig.39 and 43).

### 3. Conclusion

- (1) In chemical densified coating (CDC), it was difficult to control the thickness of the  $Y_2O_3$  coating because ceramic slurry was solidified about 10 minutes. Coating thickness attained was only about  $23\mu m$ . Many cracks were observed in the  $Y_2O_3$  coating. Therefore, atmospheric plasma spray as case 2 was selected for  $Y_2O_3$  coating. Because the atmospheric plasma spray is applicable for various materials as a coating and for thick coating.
- (2) In atmospheric plasma spray, coating thickness up to about  $60\mu m$  was attainable. The electrical resistivity of the  $Y_2O_3$  coating was  $1.0 \times 10^{11}$ ,  $5.0 \times 10^9$ ,  $1.6 \times 10^8$  and  $1.0 \times 10^7 \Omega \cdot cm$  at 200, 300, 500 and  $800^\circ C$ , respectively, which satisfy ITER design guideline. However, the  $Y_2O_3$  coating was porous and inferior in thermal shock resistivity. Peeling of about 50% of the coating was observed after 11 cycles of thermal shock test, which was performed by water quenching from  $500^\circ C$ . Adhesion strength of the  $Y_2O_3$  coating was about 40MPa.
- (3) In atmospheric plasma spray with densification treatment, Peeling occurred by difference of thermal expansion between 316SS substrate and atmospheric plasma sprayed  $Y_2O_3$  coating at densification treatment. In this method, it was unable to fabricate atmospheric plasma sprayed  $Y_2O_3$  coating with densification treatment. Therefore, in case 4, 410SS undercoating was coated on 316SS substrate to reduce peeling of the  $Y_2O_3$  coating.
- (4) In Atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment, it was able to fabricate the  $Y_2O_3$  coating that could stand 30 cycles of the thermal shock test at  $500^\circ C$ . It was considered that the thermal stress at thermal shock test was relieved by the effect of 410SS undercoating and cracks along the interface between 410SS undercoating and  $Y_2O_3$  coating. From the results of hardness test, it was confirmed that the  $Y_2O_3$  coating was densified.

From the results of trial fabrication and preliminary characterization, it can be resulted that atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment is suitable for  $Y_2O_3$  coating fabrication.

#### Acknowledgements

The technical assistance of K.Miyajima and Y.Harada (TOCALO Co. Ltd.) is gratefully acknowledged.

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Table 1 The results of the chemical analysis on 316SS substrate

| Chemical component | Analysis value (wt.%) |
|--------------------|-----------------------|
| C                  | 0.05                  |
| Si                 | 0.58                  |
| Mn                 | 1.23                  |
| P                  | 0.028                 |
| S                  | 0.003                 |
| Cr                 | 16.78                 |
| Ni                 | 10.64                 |
| Mo                 | 2.09                  |

Table 2 The results of the chemical analysis on  $Y_2O_3$  particles for ceramic slurry

| Chemical component | Analysis value (wt.%) |
|--------------------|-----------------------|
| $Y_2O_3$           | > 99.95               |
| Ave. particle size | 2.53 $\mu$ m          |

Table 3 The results of the chemical analysis on  $Y(NO_3)_3$  solution

|                           |         |
|---------------------------|---------|
| Concentration of $Y_2O_3$ | 15.17 % |
| Concentration of acid     | 4.62 N  |
| Specific gravity          | 1.385   |

Table 4 The results of the chemical analysis on  $Y_2O_3$  particles for atmospheric plasma spray  
(particle size : 10~45 $\mu$ m)

| Chemical component | Analysis value (wt.%) |
|--------------------|-----------------------|
| $Al_2O_3$          | 0.02                  |
| $SiO_2$            | 0.02                  |
| $Fe_2O_3$          | 0.01                  |
| $Y_2O_3$           | 99.95                 |



Table 5 Atmospheric plasma spray conditions for  $Y_2O_3$  coating

| Material                      | $Y_2O_3$ particles<br>(particle size : 10~45 $\mu$ m) |                      |
|-------------------------------|-------------------------------------------------------|----------------------|
| Plasma gas                    | Ar                                                    | H <sub>2</sub>       |
| Flow rate (m <sup>3</sup> /s) | 7.0x10 <sup>-4</sup>                                  | 2.0x10 <sup>-4</sup> |
| Plasma current (A)            | 550                                                   |                      |
| Plasma voltage (V)            | 74                                                    |                      |
| Spray distance (mm)           | 120                                                   |                      |

Table 6 The results of the micro Vickers hardness test of the  $Y_2O_3$  coating by atmospheric plasma spray (case 2)

| Test No. | Hardness (Hv) |
|----------|---------------|
| 1        | 464           |
| 2        | 478           |
| 3        | 357           |
| 4        | 297           |
| 5        | 405           |
| 6        | 405           |
| 7        | 251           |
| 8        | 360           |
| 9        | 468           |
| 10       | 401           |
| Average  | 389           |

Table 7 The results of the adhesion test of the  $Y_2O_3$  coating by atmospheric plasma spray (case 2)

| Test No. | Adhesion (MPa) |
|----------|----------------|
| 1        | 40.1           |
| 2        | 38.9           |
| 3        | 42.1           |
| Average  | 40.4           |

Table 8 The results of the electrical resistivity of the  $Y_2O_3$  coating by atmospheric plasma spray (case 2)

| Temperature (°C) | Electrical resistivity ( $\Omega \cdot \text{cm}$ ) |
|------------------|-----------------------------------------------------|
| 25               | $3.1 \times 10^{14}$                                |
| 100              | $1.3 \times 10^{13}$                                |
| 200              | $1.0 \times 10^{11}$                                |
| 300              | $5.0 \times 10^9$                                   |
| 500              | $1.6 \times 10^8$                                   |
| 800              | $1.0 \times 10^7$                                   |
| 900              | $5.0 \times 10^6$                                   |

Table 9 The results of the chemical analysis on 410SS particles for undercoating (particle size : 10~74 $\mu\text{m}$ )

| Chemical component | Analysis value (wt.%) |
|--------------------|-----------------------|
| C                  | 0.012                 |
| Si                 | 0.83                  |
| Mn                 | 0.15                  |
| P                  | 0.025                 |
| S                  | 0.001                 |
| Cr                 | 12.57                 |
| Ni                 | 0.11                  |

Table 10 Atmospheric plasma spray conditions for 410SS undercoating

| Material                      | 410SS particles<br>(particle size : 10~74 $\mu\text{m}$ ) |                      |
|-------------------------------|-----------------------------------------------------------|----------------------|
| Plasma gas                    | Ar                                                        | H <sub>2</sub>       |
| Flow rate (m <sup>3</sup> /s) | $9.2 \times 10^{-4}$                                      | $1.5 \times 10^{-4}$ |
| Plasma current (A)            | 550                                                       |                      |
| Plasma voltage (V)            | 72                                                        |                      |
| Spray distance (mm)           | 130                                                       |                      |

Table 11 The results of the micro Vickers hardness test of the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4)

| Test No. | Hardness (Hv) |
|----------|---------------|
| 1        | 579           |
| 2        | 503           |
| 3        | 493           |
| 4        | 530           |
| 5        | 542           |
| 6        | 508           |
| 7        | 554           |
| 8        | 429           |
| 9        | 503           |
| 10       | 542           |
| Average  | 518           |

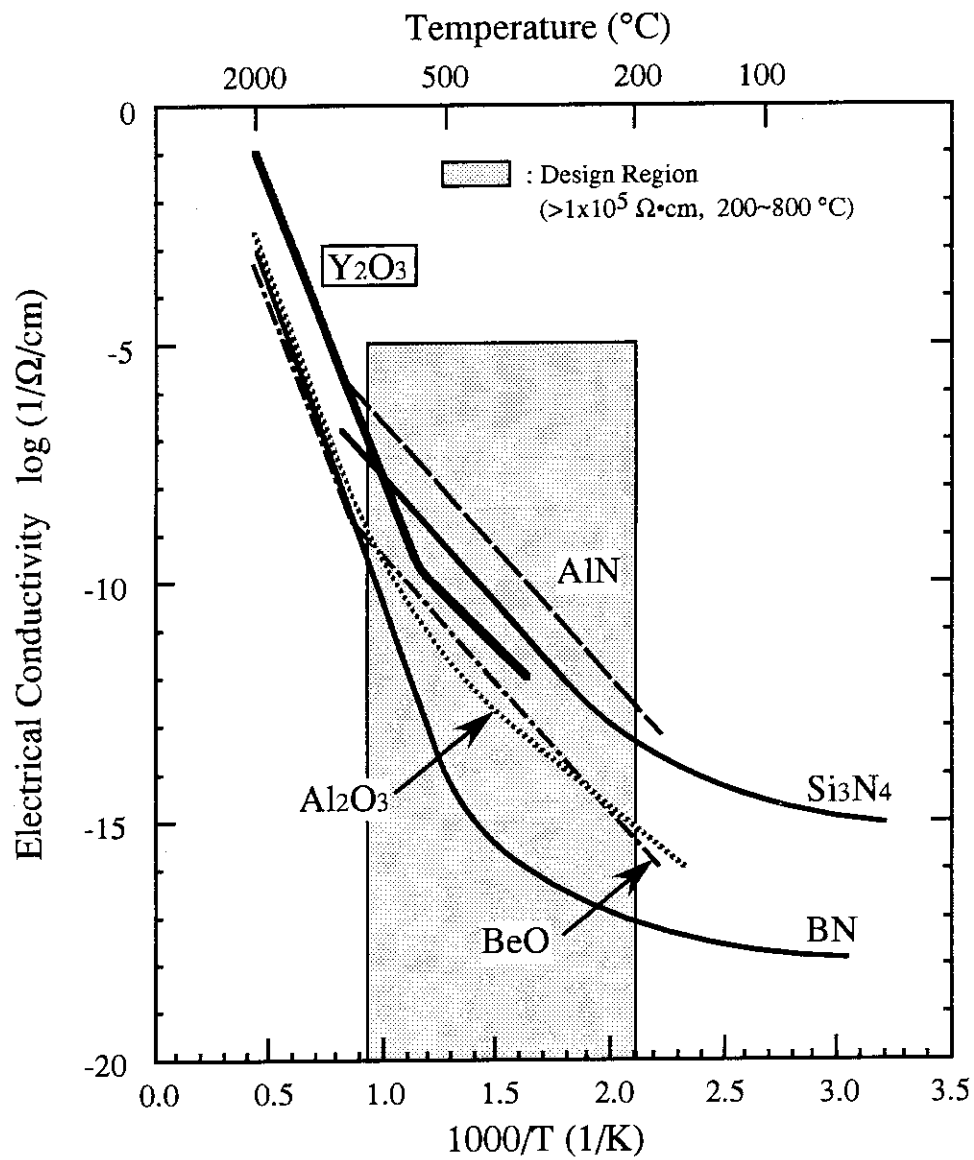


Fig.1 Electrical conductivity vs. temperature with different electrical insulator  
(A.A.Bauer and J.L.Bates, Battelle Mem. Inst. Rept., 1930, (Jul. 31, 1974))

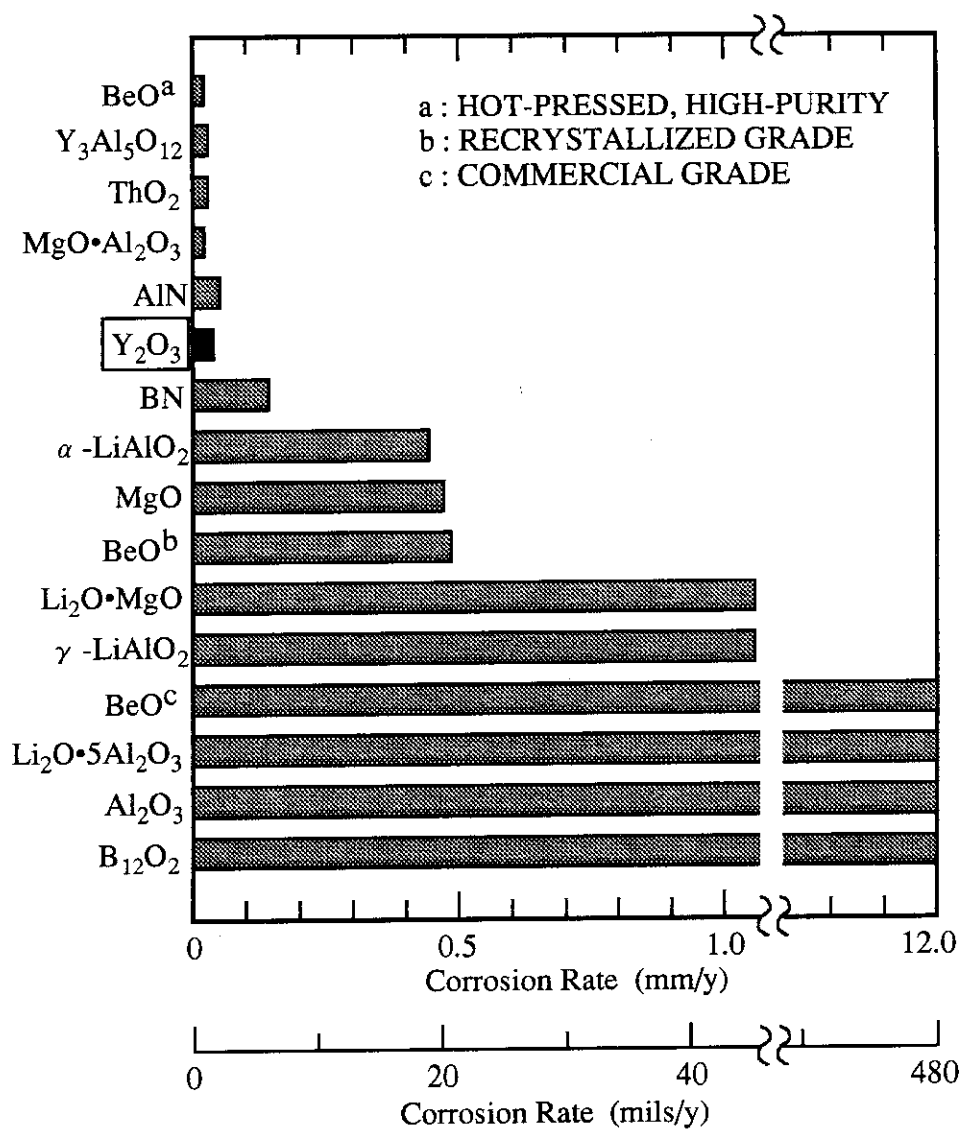


Fig.2 Corrosion rate of insulating materials by lithium at 375°C  
(E.J.Cairns et al., in Development of High Energy Batteries for Electric Vehicles,  
Progress Report for the Period July 1970-June 1971, p.59 USAEC report ANL-7888  
(Dec. 1971))

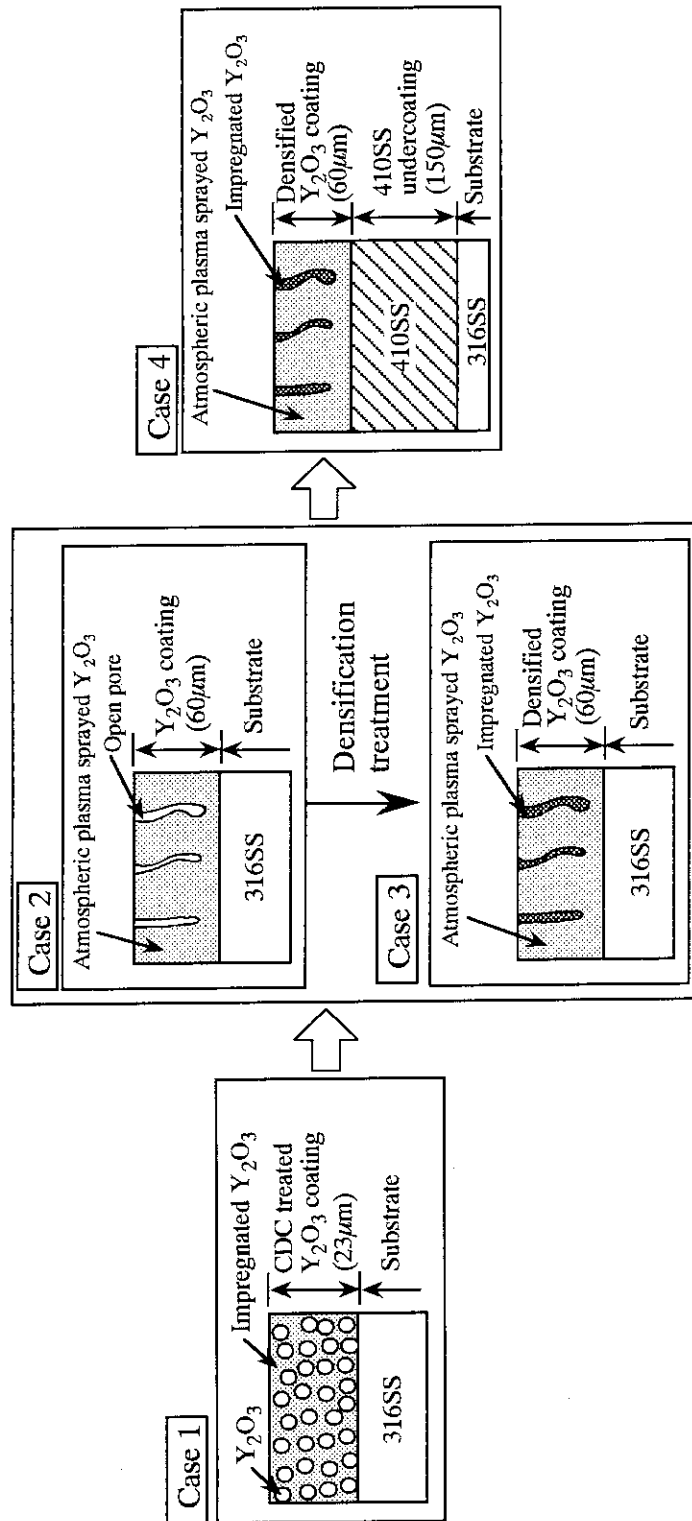


Fig.3 Technical flow of trial fabrication of  $Y_2O_3$  coating

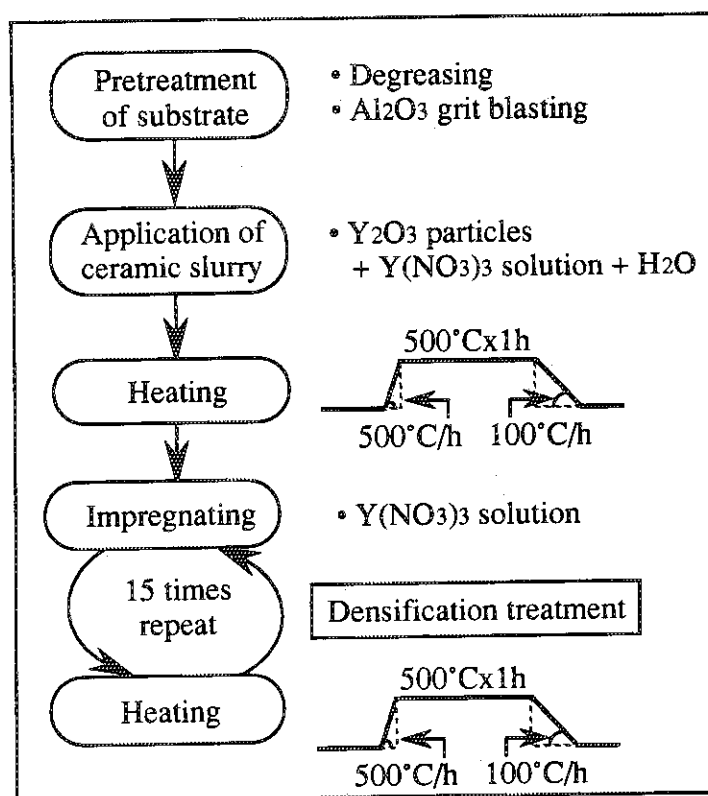


Fig.4 The fabrication process in chemical densified coating (case 1)

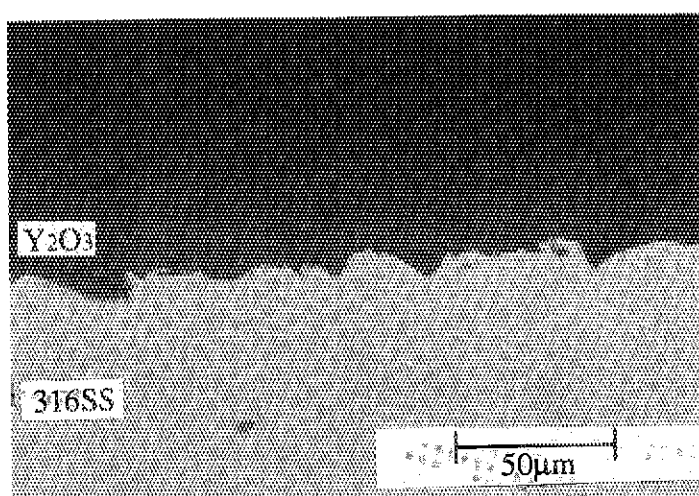


Fig.5 Optical microscope photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by chemical densified coating (case 1)

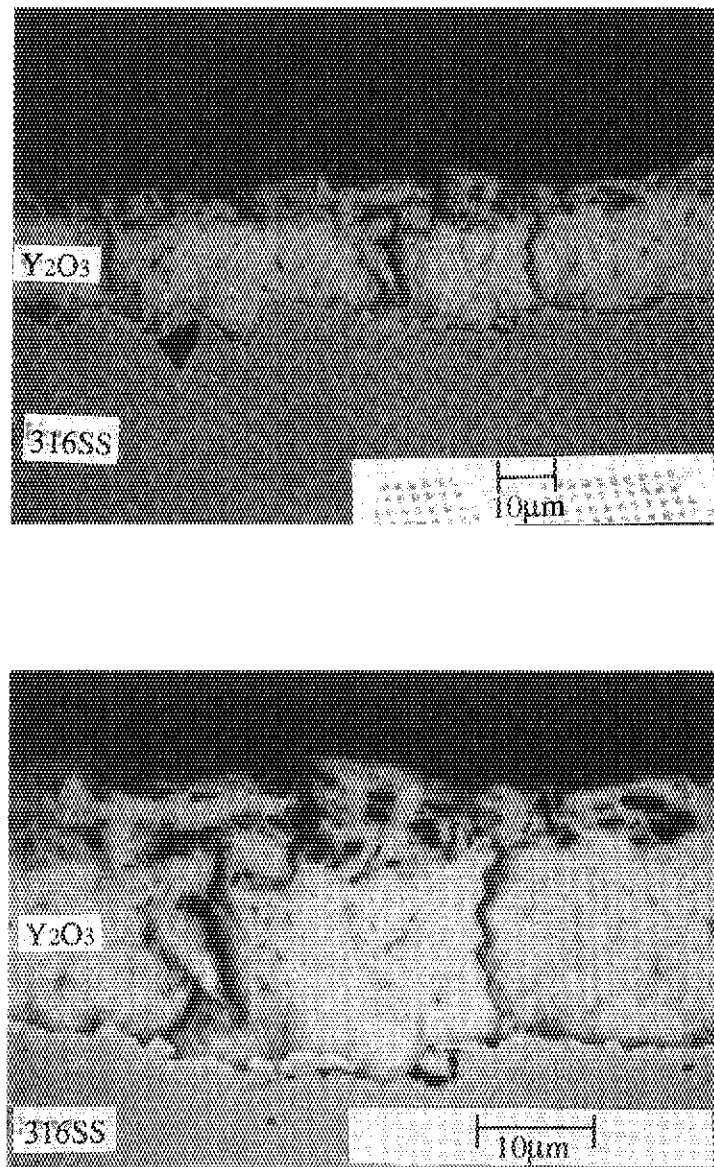


Fig.6 Scanning electron microscope (SEM) photograph of a cross section in the  $\text{Y}_2\text{O}_3$  coating by chemical densified coating (case 1)



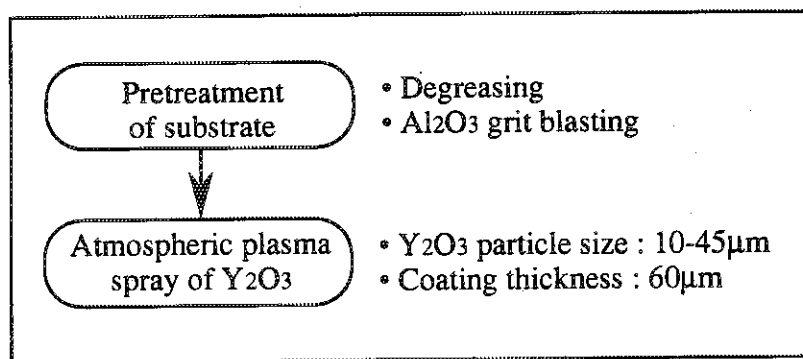


Fig.7 The fabrication process in atmospheric plasma spray (case 2)

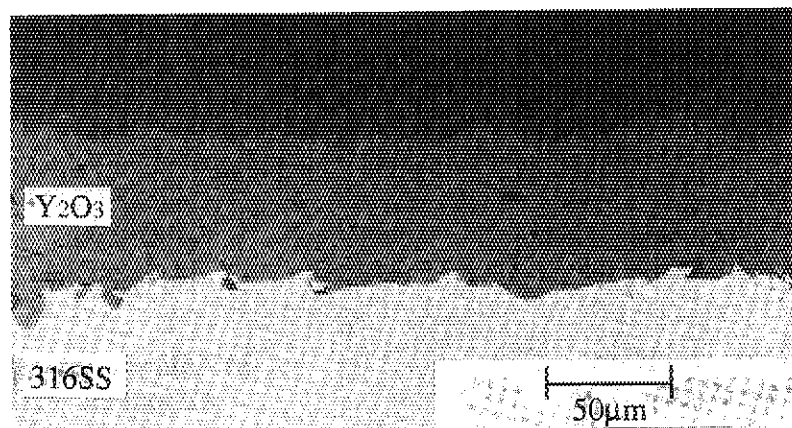


Fig.8 Optical microscope photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2)

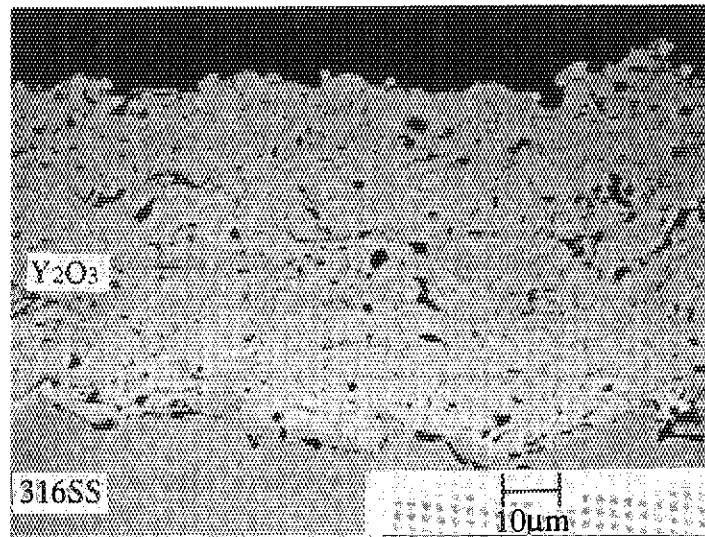


Fig.9 Scanning electron microscope (SEM) photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray (case 2)

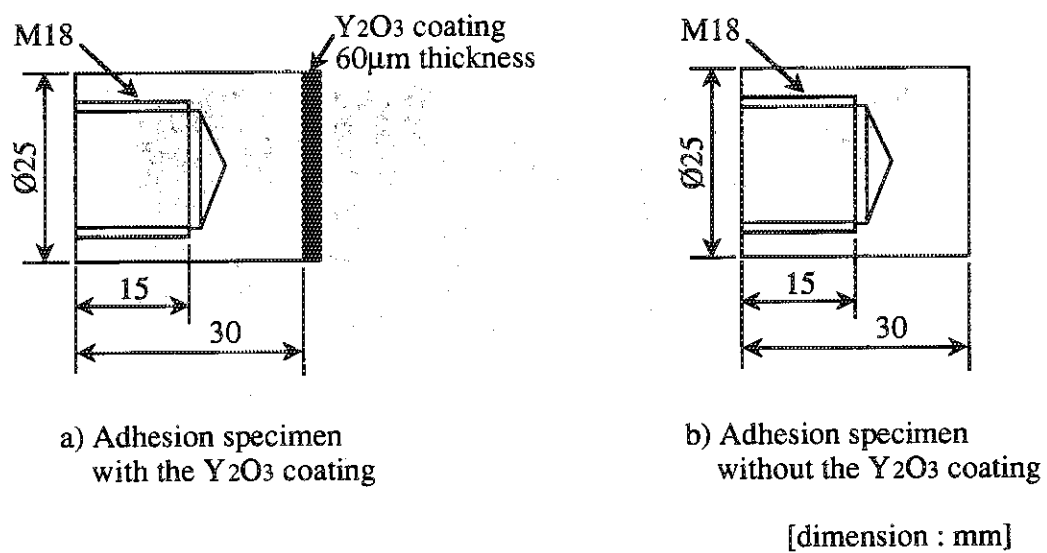


Fig.10 Adhesion specimens

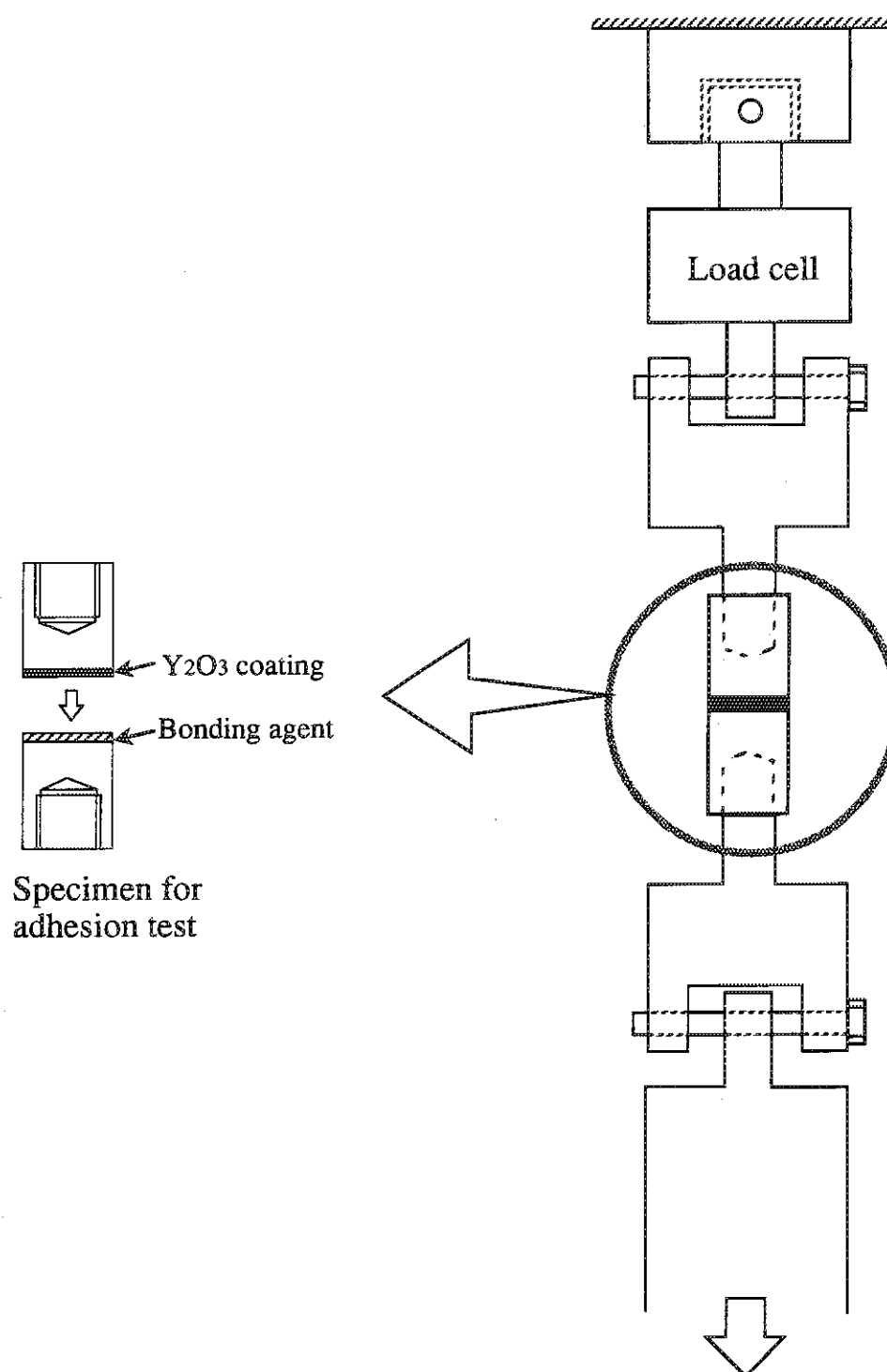
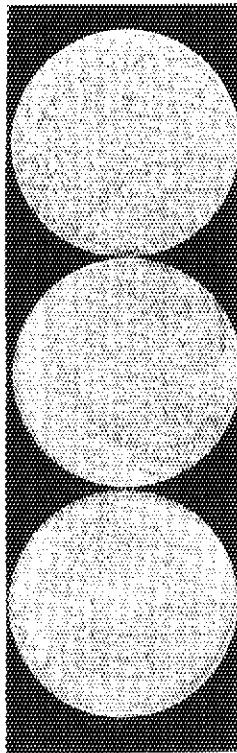
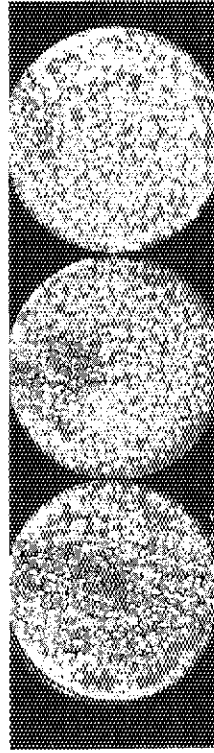


Fig.11 Outline of adhesion test



a) The side of specimen with the Y<sub>2</sub>O<sub>3</sub> coating



b) The side of specimen without the Y<sub>2</sub>O<sub>3</sub> coating

10mm

Fig.12 Appearance photographs of the surface of specimens with the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) and without Y<sub>2</sub>O<sub>3</sub> coating after adhesion test

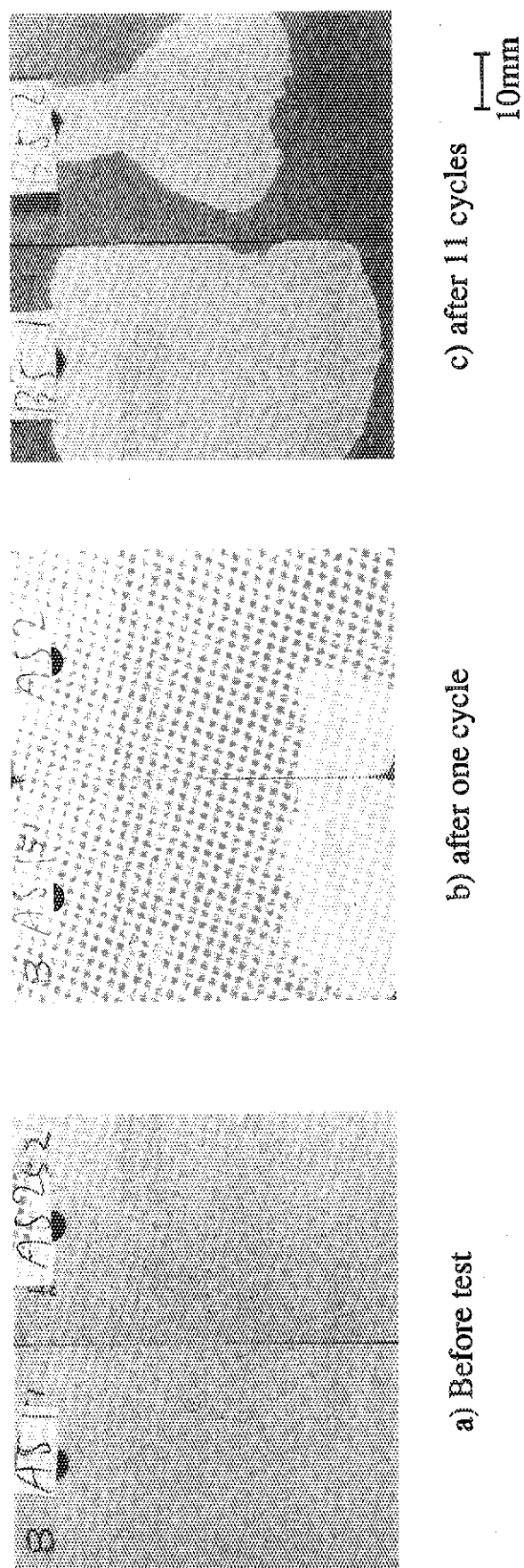


Fig. 13 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray (case 2) before and after thermal shock test at  $500^\circ C$

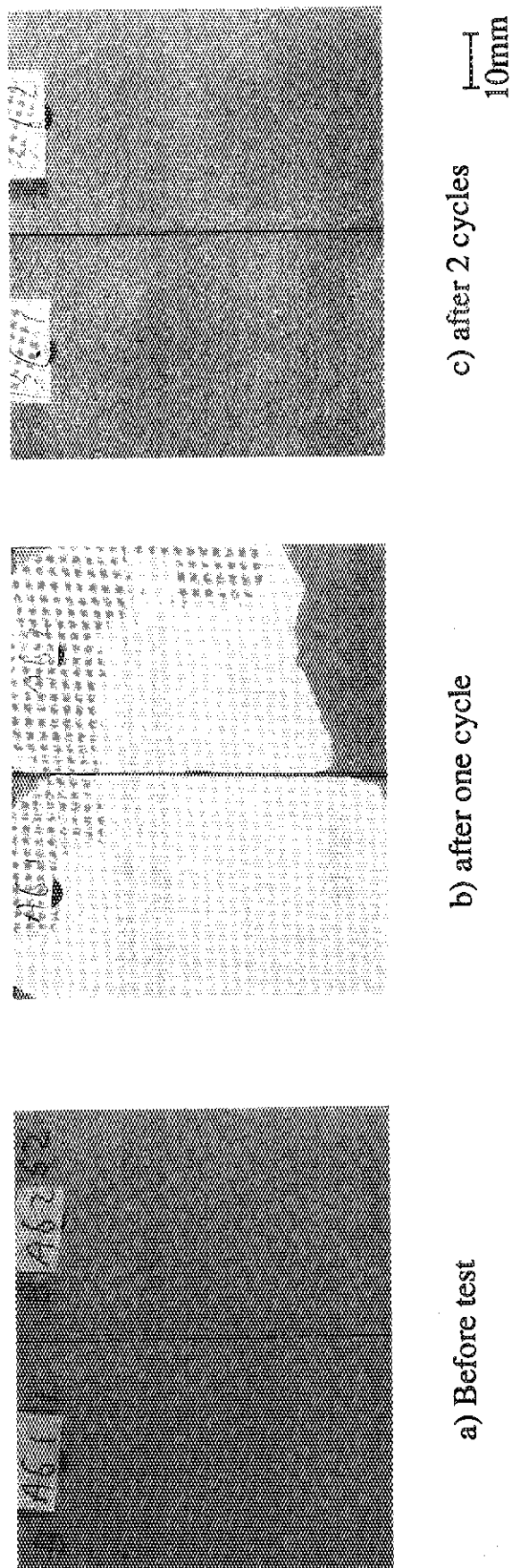


Fig.14 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray (case 2) before and after thermal shock test at  $600^\circ C$

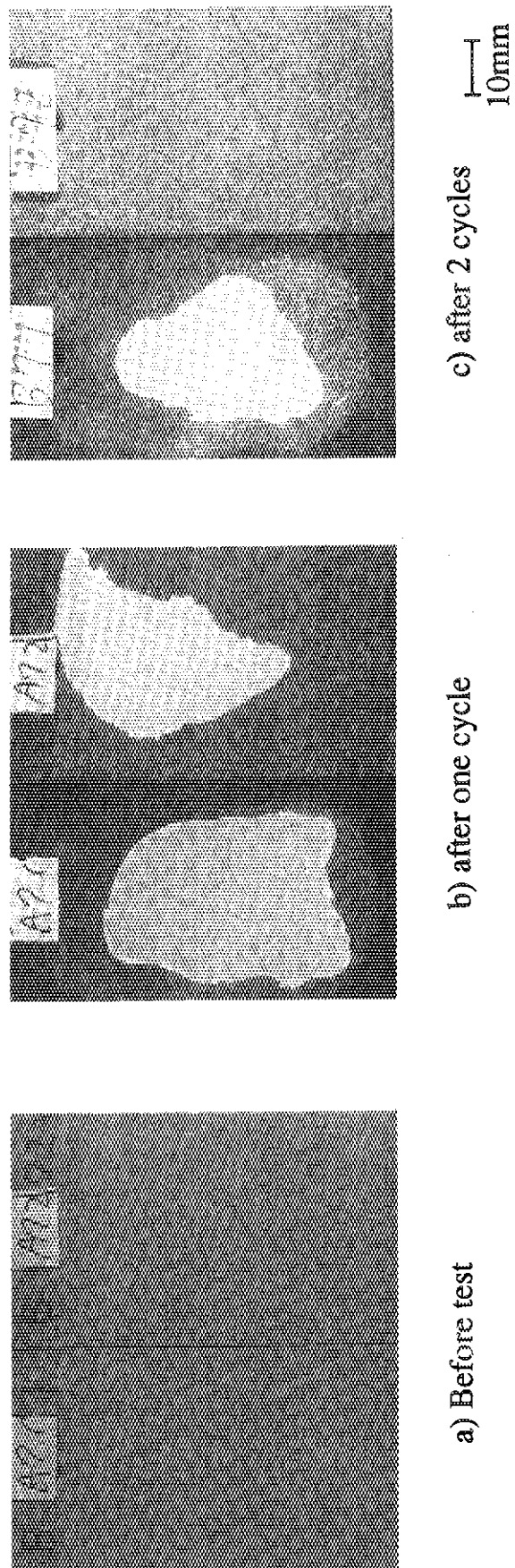


Fig.15 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray (case 2) before and after thermal shock test at 700°C

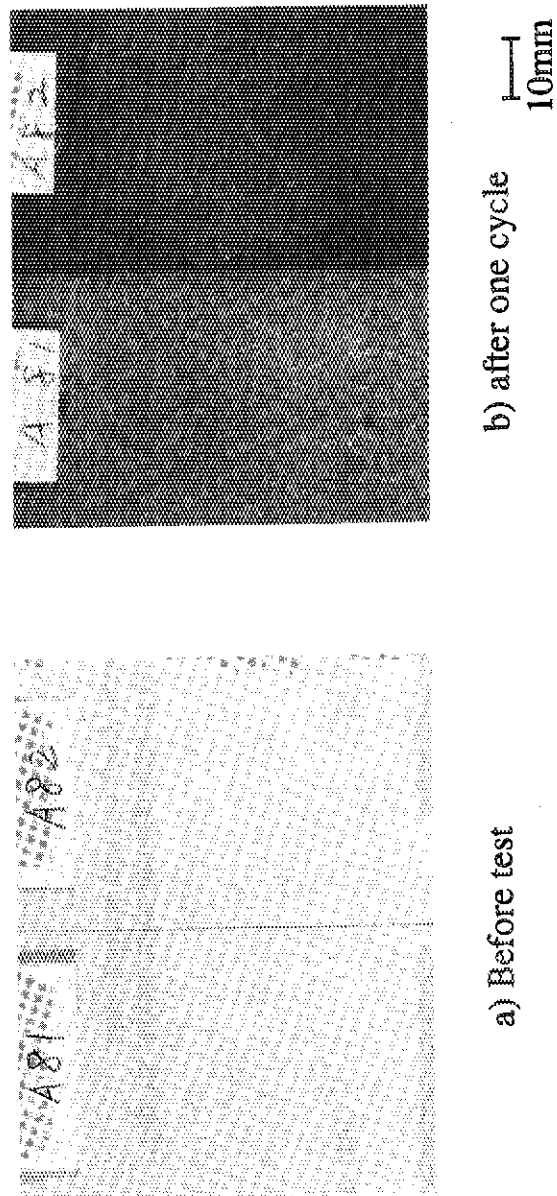


Fig.16 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray (case 2) before and after thermal shock test at 800°C



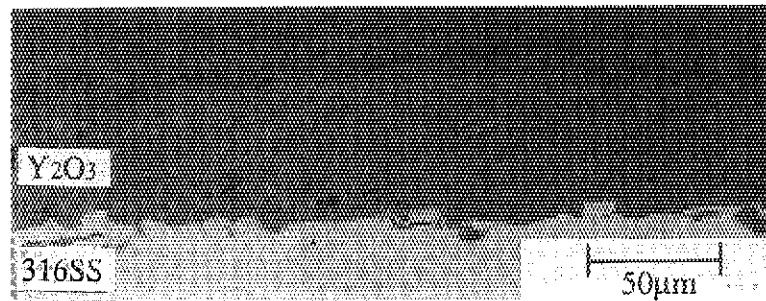


Fig.17 Optical microscope photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after 11 cycles of thermal shock test at 500°C

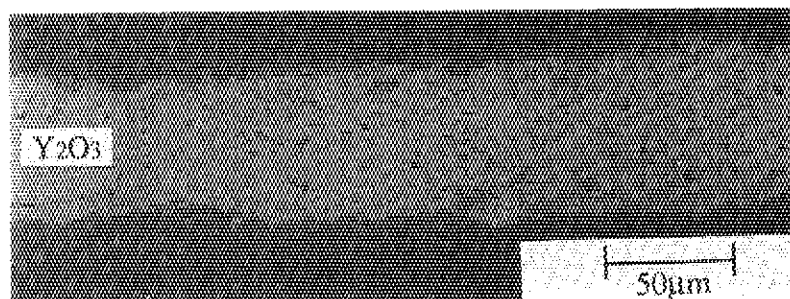


Fig.18 Optical microscope photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after 2 cycles of thermal shock test at 600°C

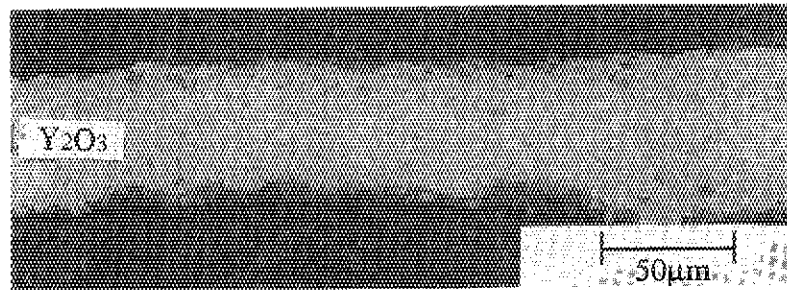


Fig.19 Optical microscope photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after 2 cycles of thermal shock test at 700°C

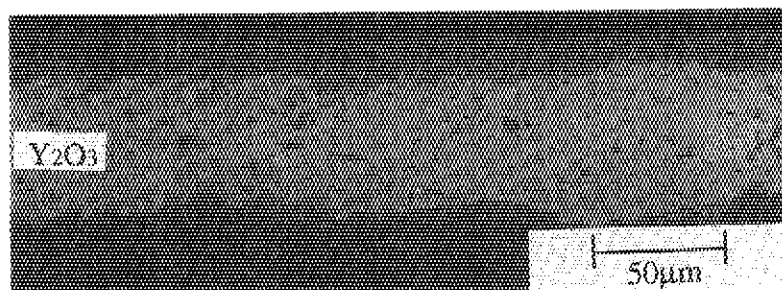


Fig.20 Optical microscope photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after one cycle of thermal shock test at 800°C

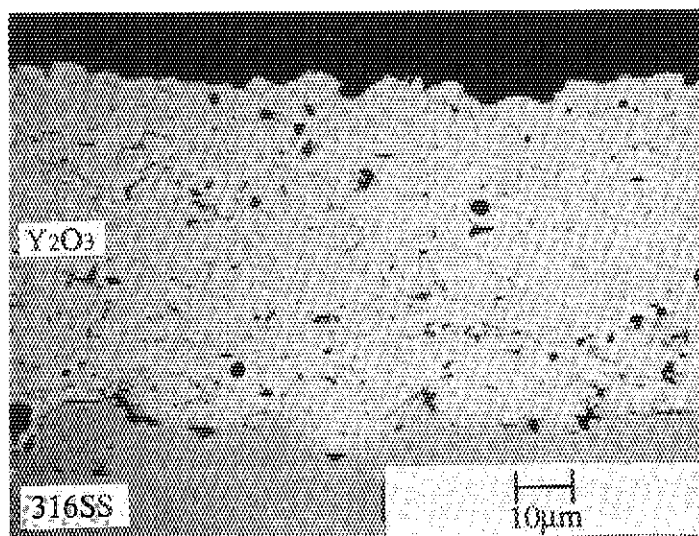


Fig.21 Scanning electron microscope (SEM) photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after 11 cycles of thermal shock test at 500°C

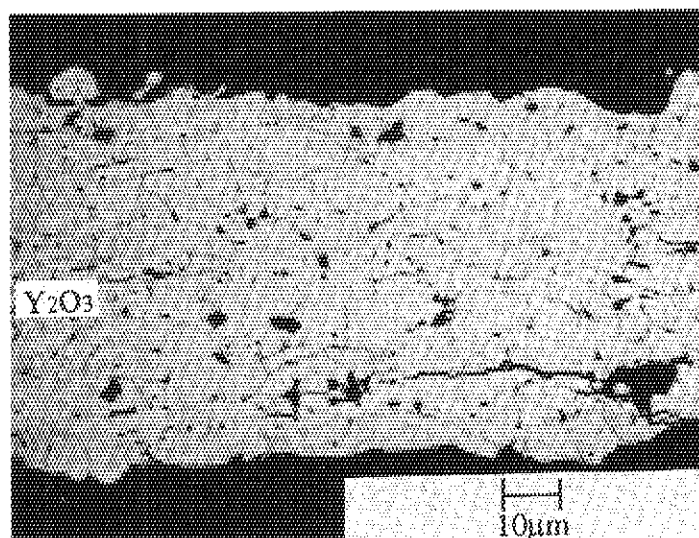


Fig.22 Scanning electron microscope (SEM) photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after 2 cycles of thermal shock test at 600°C

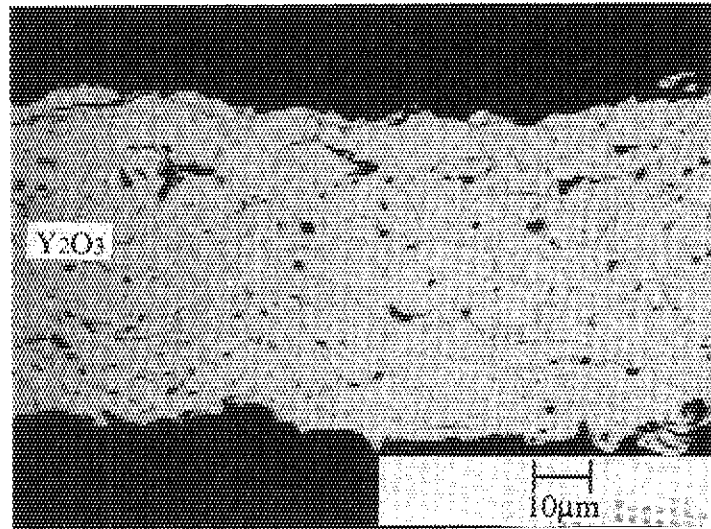


Fig.23 Scanning electron microscope (SEM) photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after 2 cycles of thermal shock test at 700°C

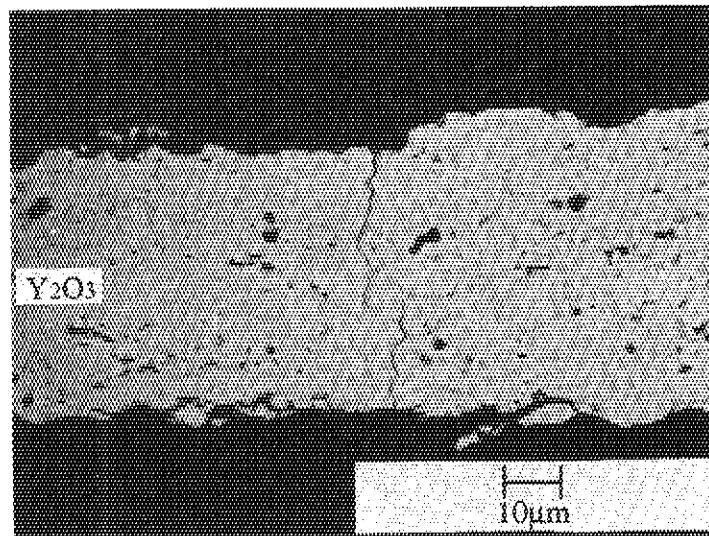


Fig.24 Scanning electron microscope (SEM) photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) after one cycle of thermal shock test at 800°C

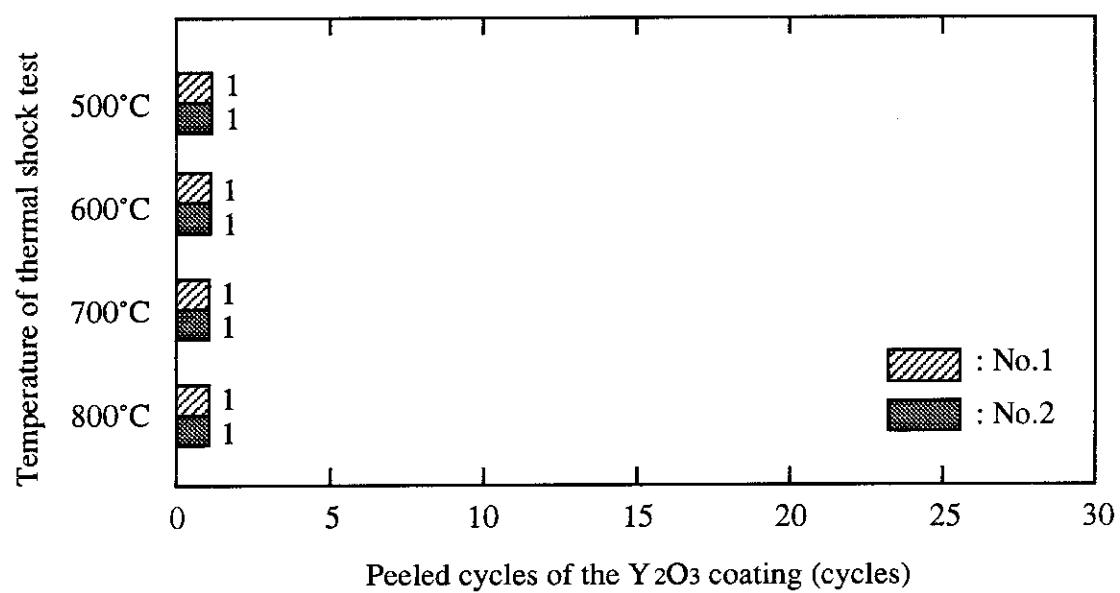


Fig.25 Peeled cycles of the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray (case 2) in the thermal shock test

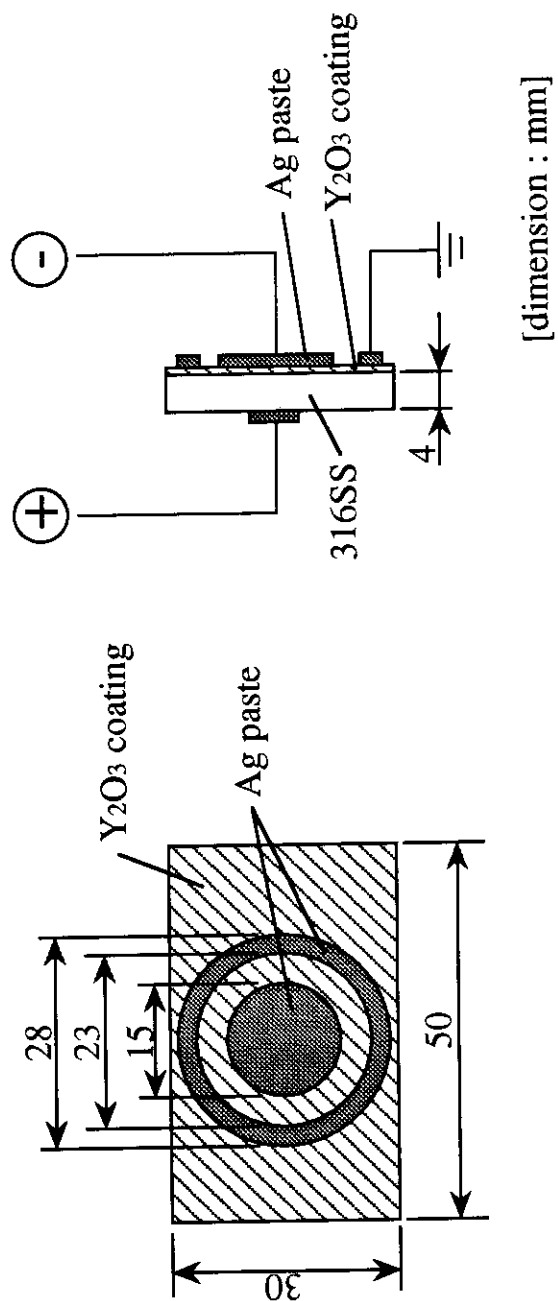


Fig.26 Specimen and outline for electrical resistivity measurement of the  $Y_2O_3$  coating

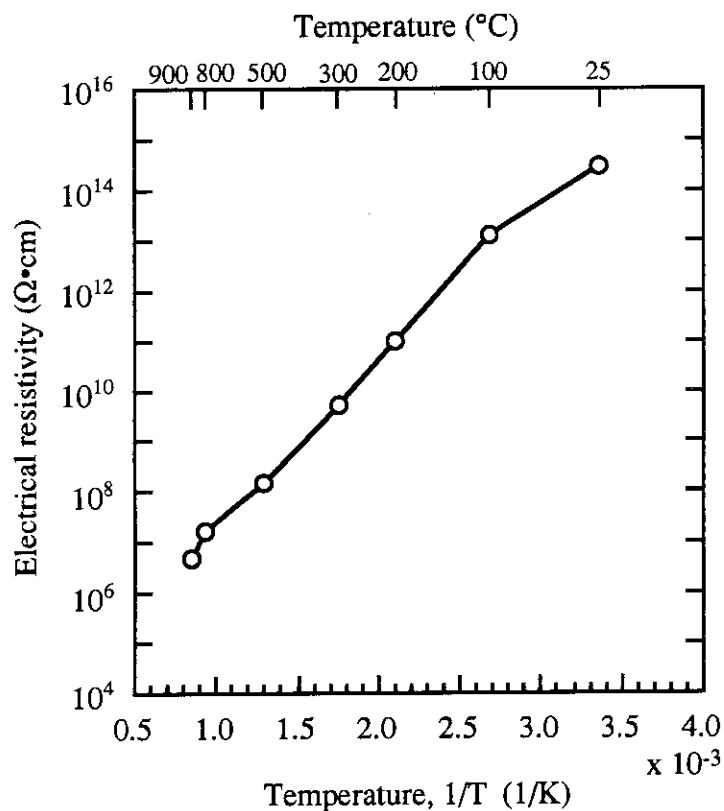


Fig.27 Electrical resistivity of the  $\text{Y}_2\text{O}_3$  coating by atmospheric plasma spray (case 2)

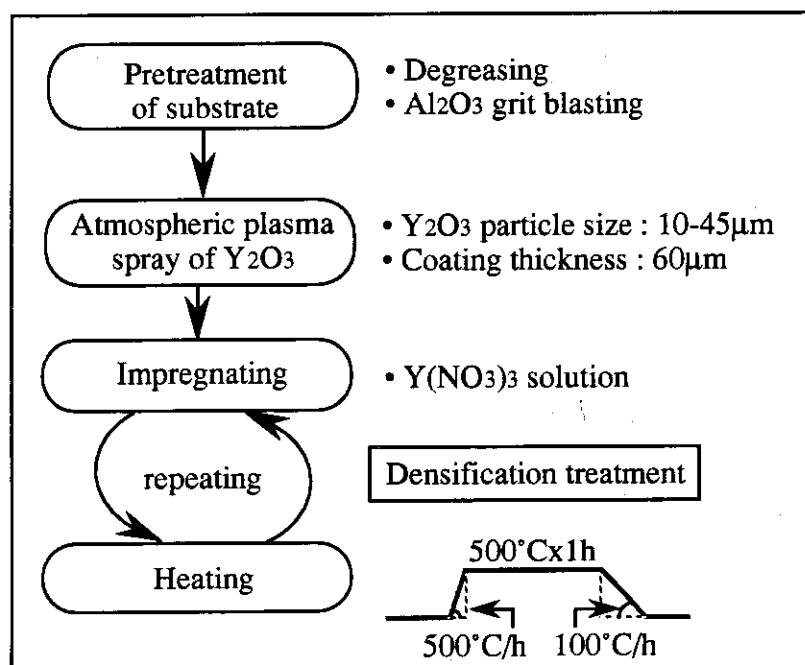


Fig.28 The fabrication process in atmospheric plasma spray with densification treatment (case 3)

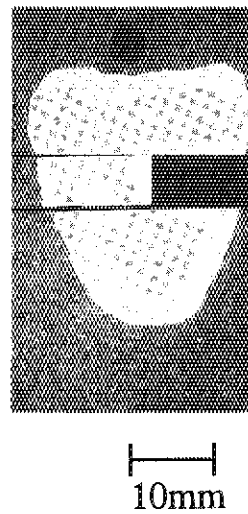


Fig.29 Appearance photograph of the surface of specimen with the  $Y_2O_3$  coating by atmospheric plasma spray with densification treatment (case 3)

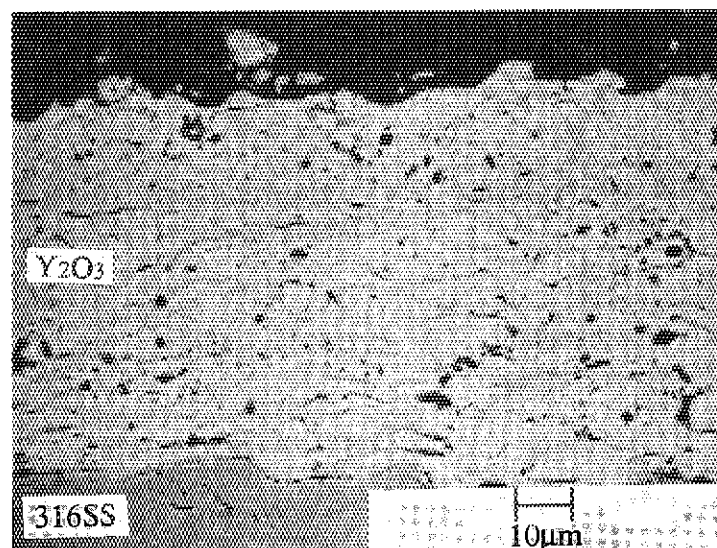


Fig.30 Scanning electron microscope (SEM) photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray with densification treatment (case 3)



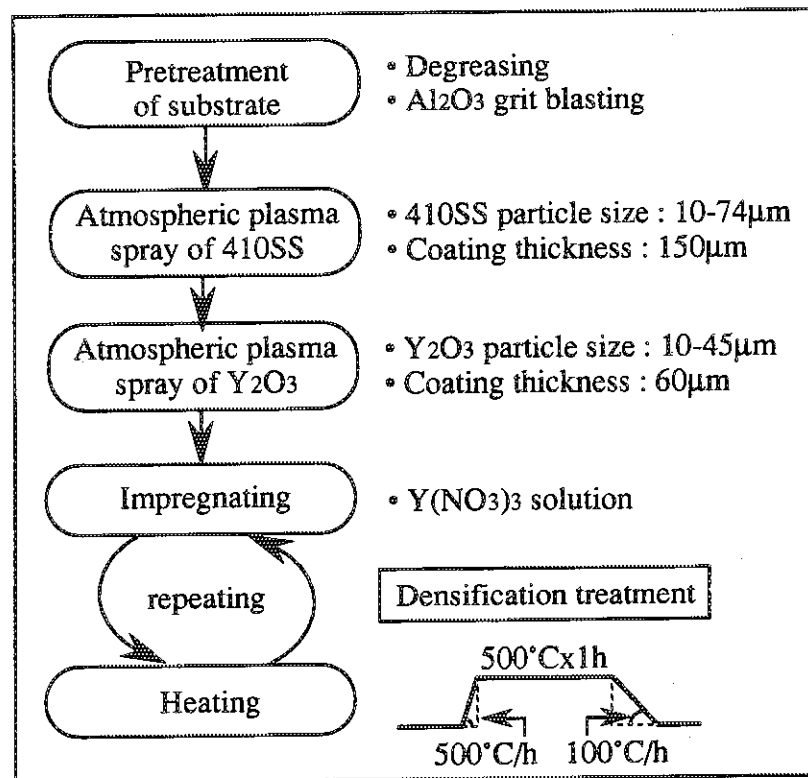


Fig.31 The fabrication process in atmospheric plasma spray undercoated by 410SS between 316SS substrate and Y<sub>2</sub>O<sub>3</sub> coating with densification treatment (case 4)

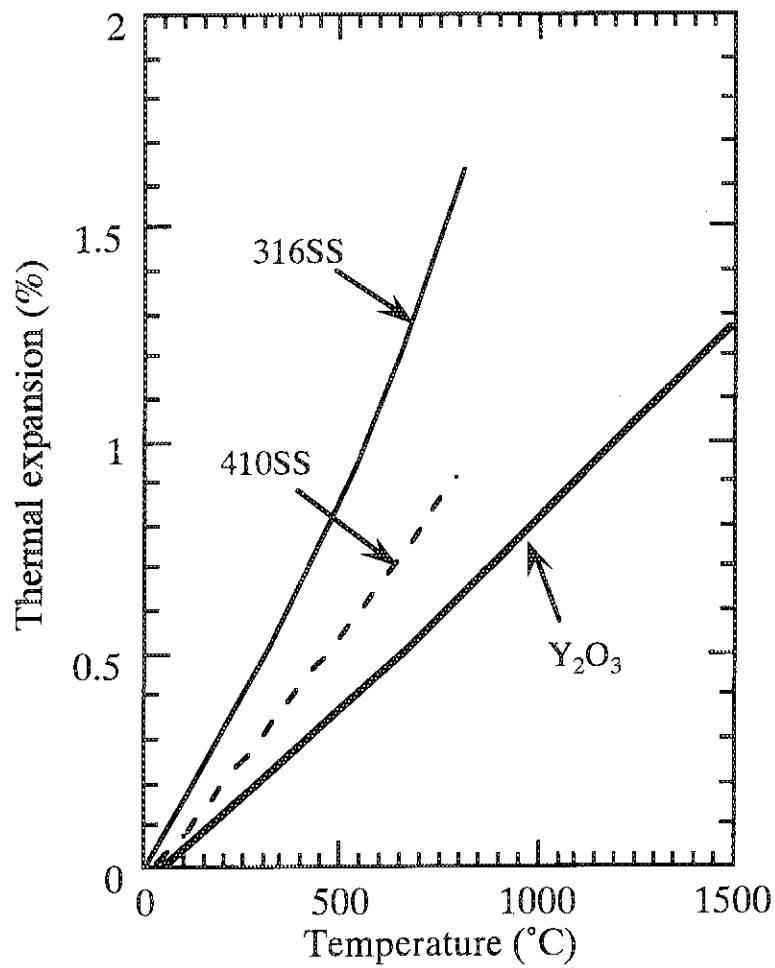


Fig.32 Thermal expansion of 316SS, 410SS and Y<sub>2</sub>O<sub>3</sub>

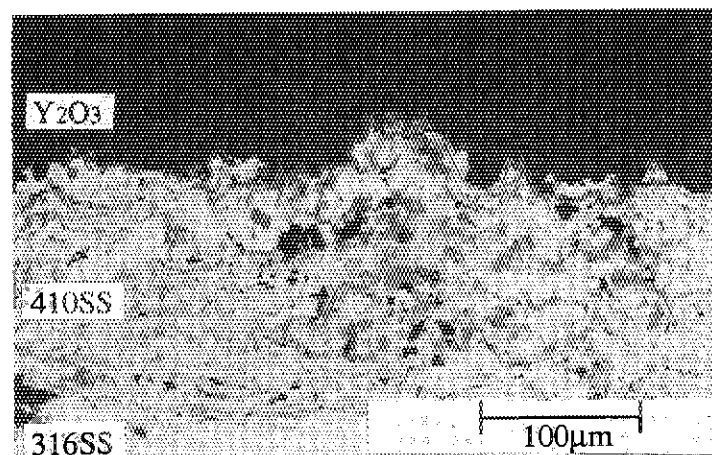


Fig.33 Optical microscope photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and Y<sub>2</sub>O<sub>3</sub> coating with densification treatment (case 4)

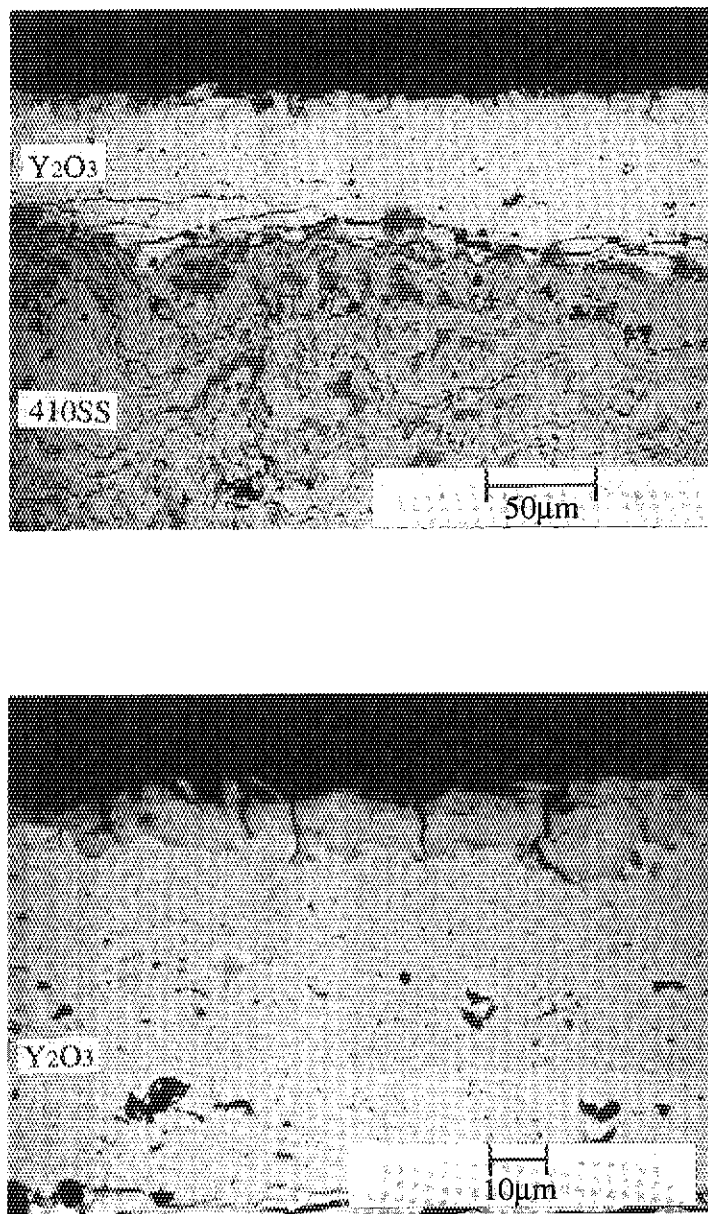


Fig.34 Scanning electron microscope (SEM) photographs of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4)

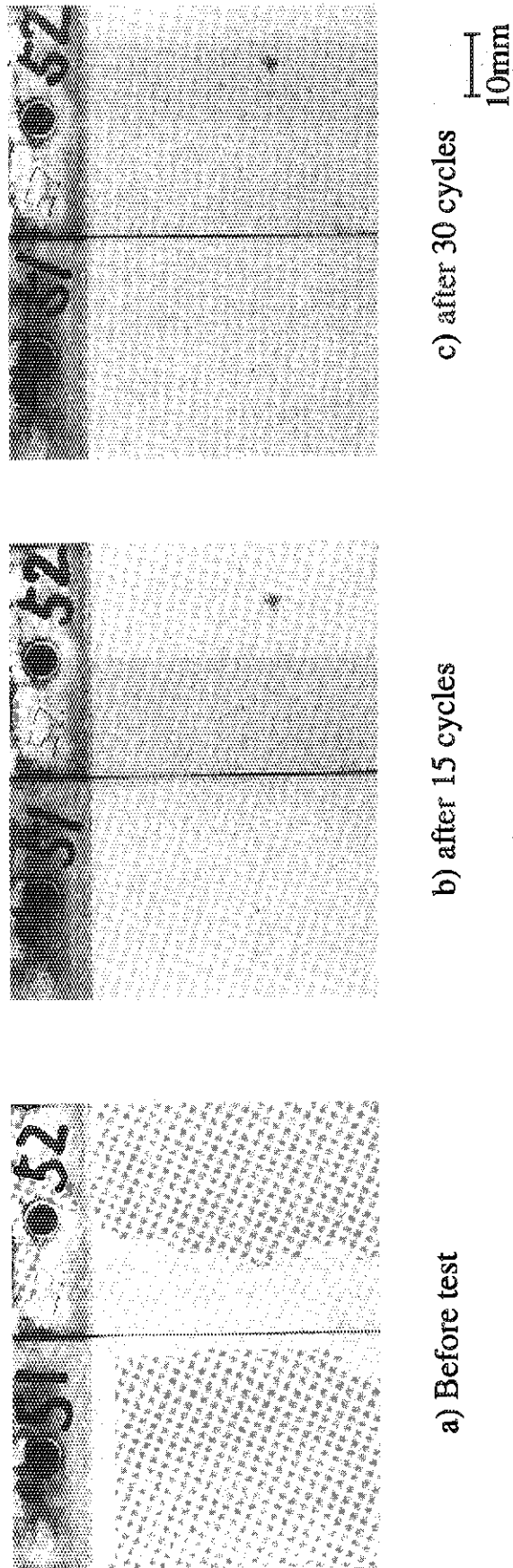


Fig.35 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) before and after thermal shock test at  $500^\circ\text{C}$

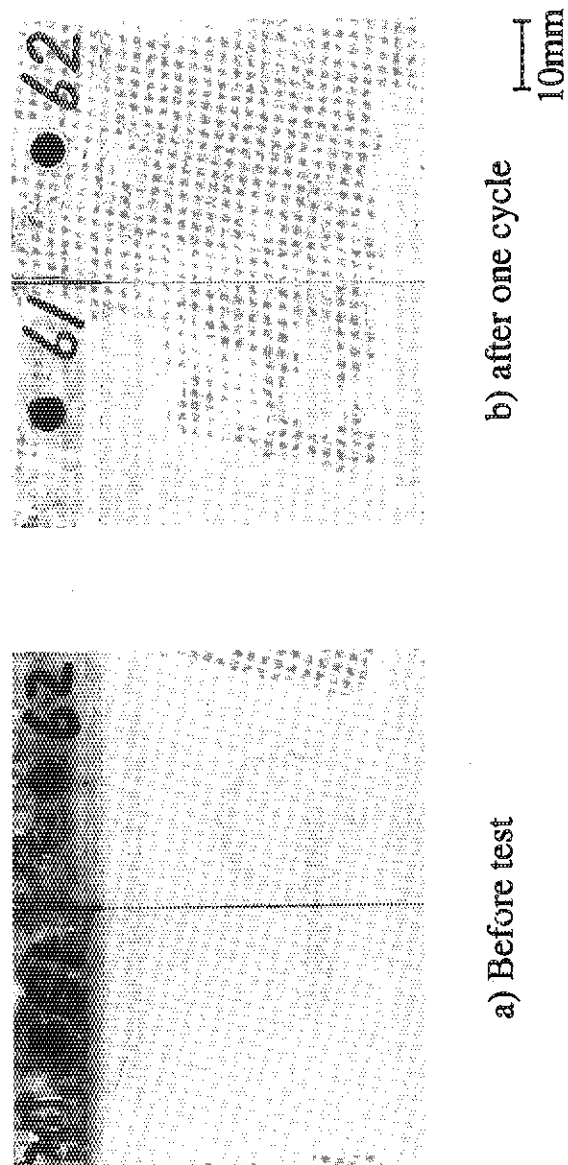


Fig.36 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) before and after thermal shock test at  $600^\circ C$

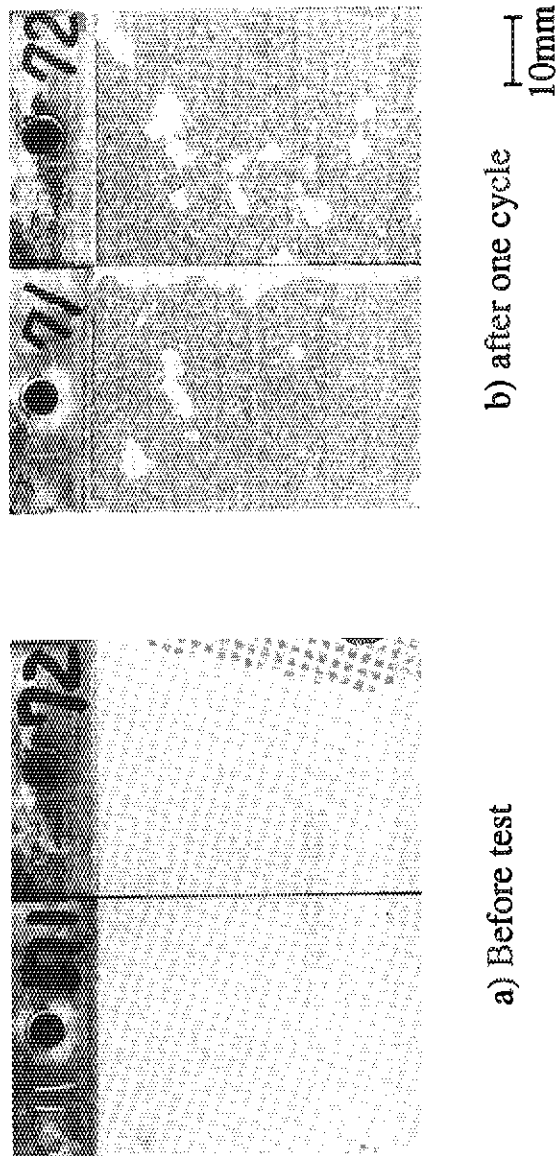


Fig.37 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) before and after thermal shock test at  $700^\circ C$

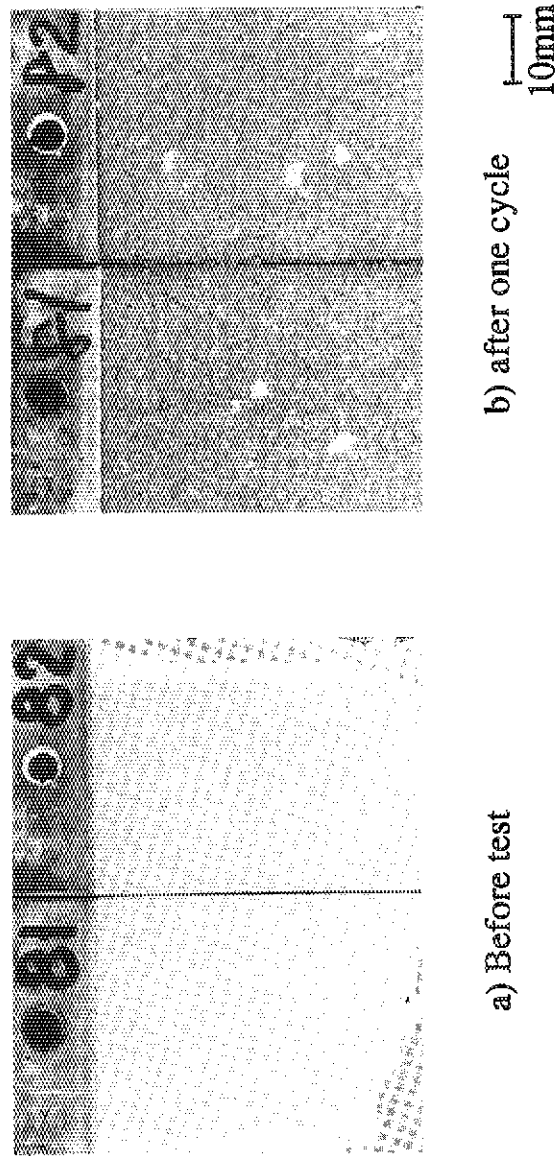


Fig.38 Appearance photographs of the surface of specimens with the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) before and after thermal shock test at  $800^\circ C$

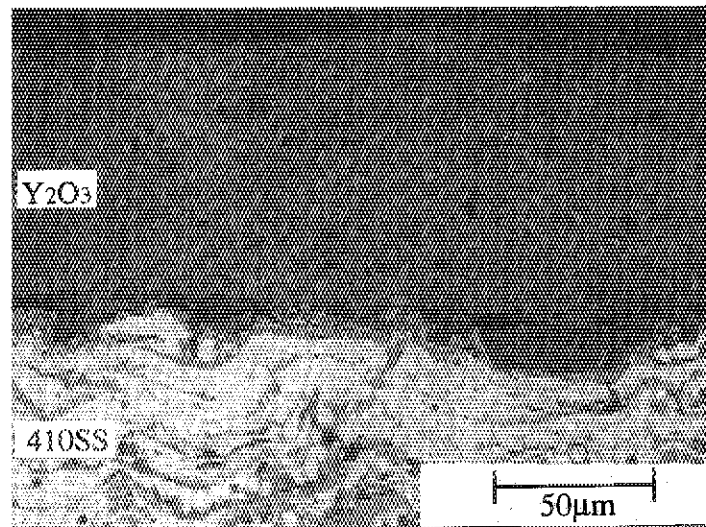


Fig.39 Optical microscope photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) after 30 cycles of thermal shock test at 500°C

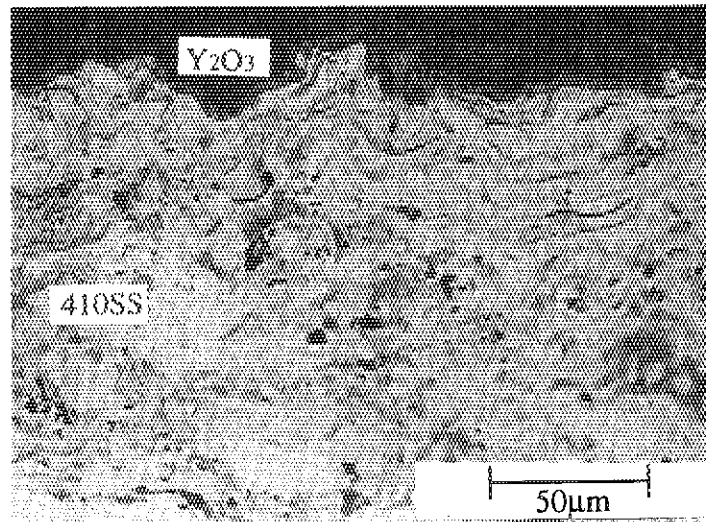


Fig.40 Optical microscope photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) after one cycle of thermal shock test at 600°C



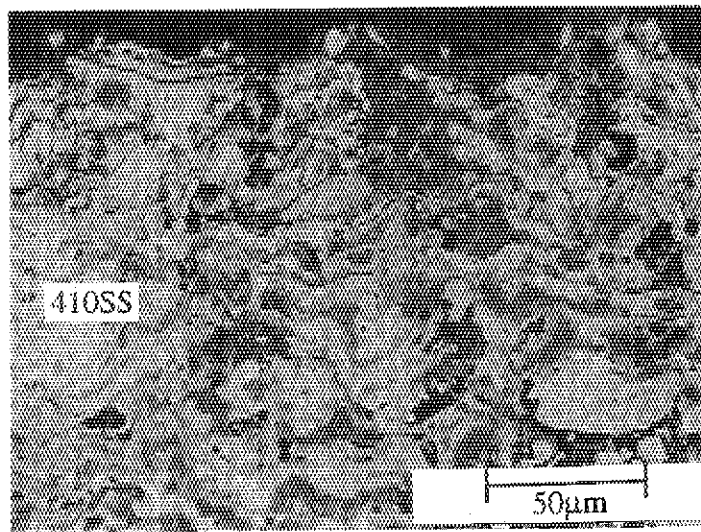


Fig.41 Optical microscope photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) after one cycle of thermal shock test at 700°C

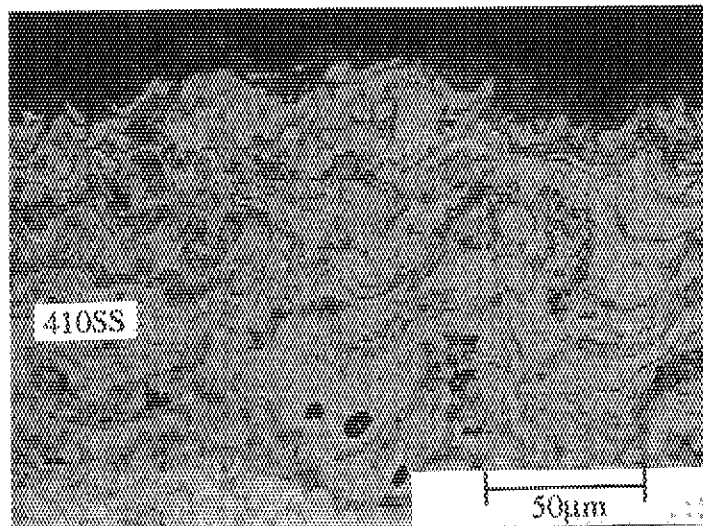


Fig.42 Optical microscope photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) after one cycle of thermal shock test at 800°C

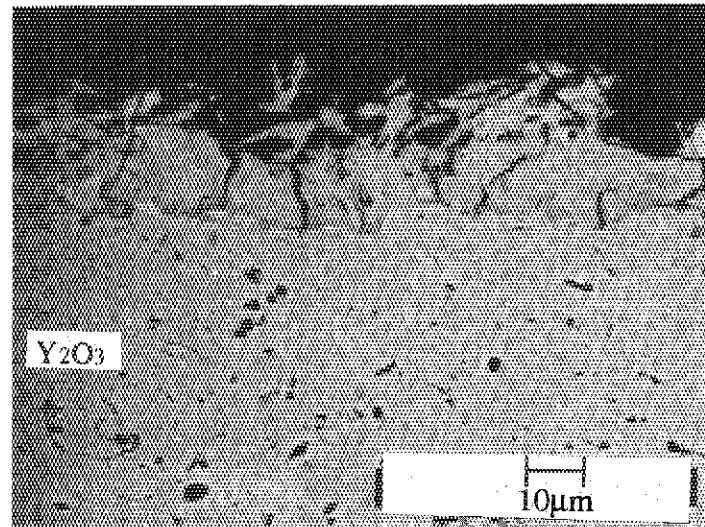


Fig.43 Scanning electron microscope (SEM) photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and Y<sub>2</sub>O<sub>3</sub> coating with densification treatment (case 4) after 30 cycles of thermal shock test at 500°C

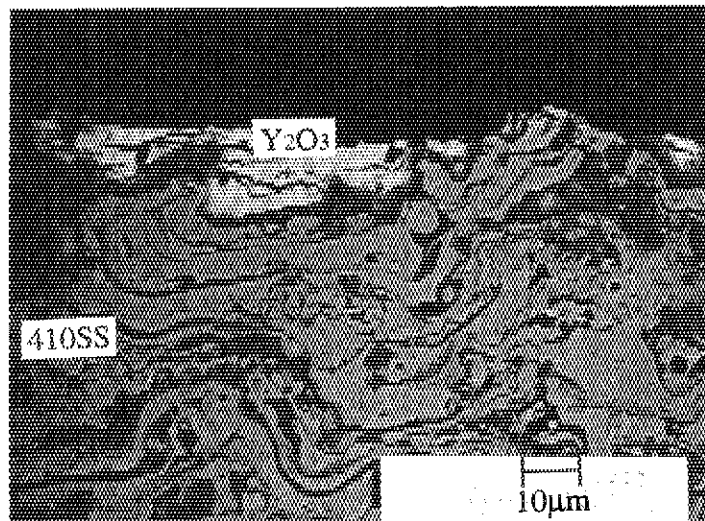


Fig.44 Scanning electron microscope (SEM) photograph of a cross section in the Y<sub>2</sub>O<sub>3</sub> coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and Y<sub>2</sub>O<sub>3</sub> coating with densification treatment (case 4) after one cycle of thermal shock test at 600°C

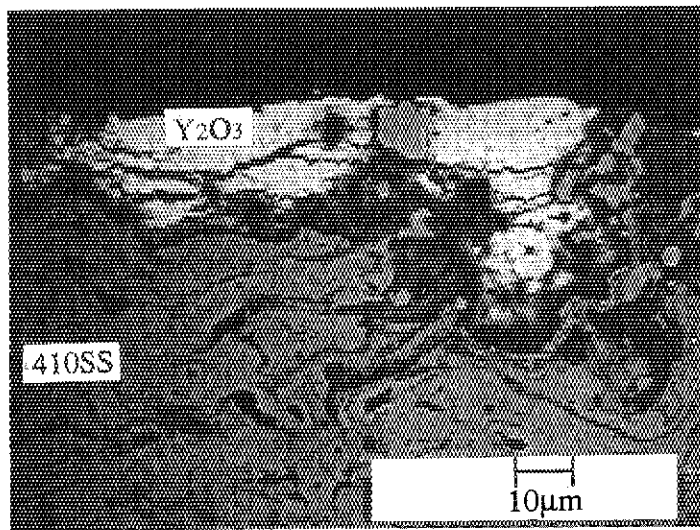


Fig.45 Scanning electron microscope (SEM) photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) after one cycle of thermal shock test at  $700^\circ C$

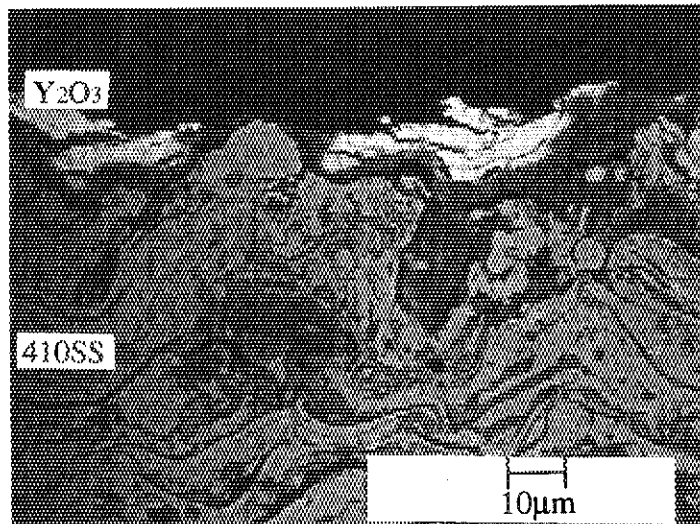
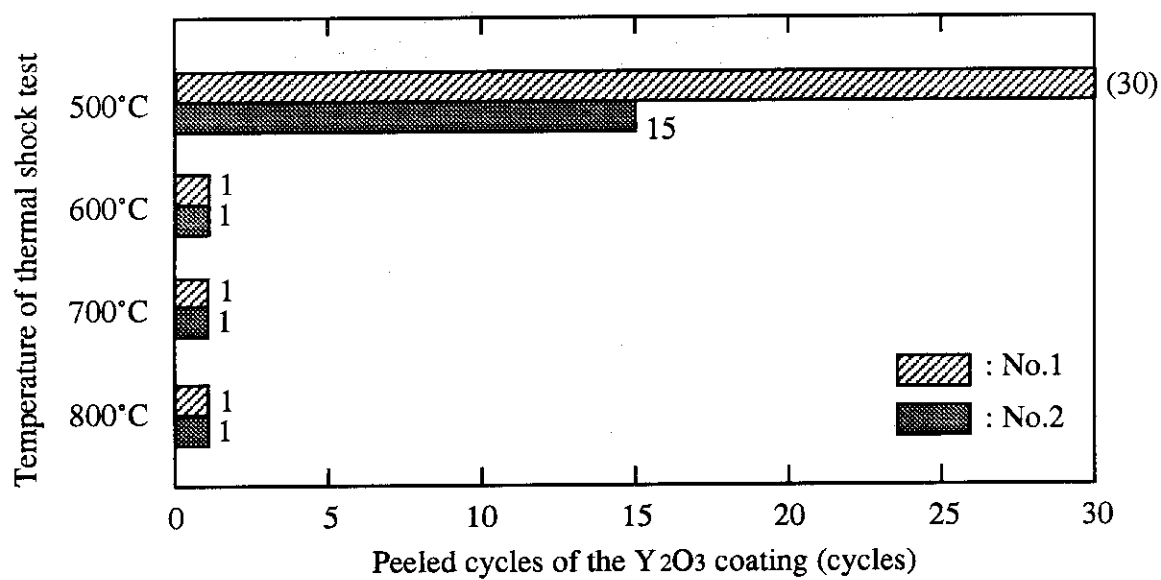


Fig.46 Scanning electron microscope (SEM) photograph of a cross section in the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) after one cycle of thermal shock test at  $800^\circ C$



\* (30) is shown that  $Y_2O_3$  coating was sound after 30 cycles.

Fig.47 Peeled cycles of the  $Y_2O_3$  coating by atmospheric plasma spray undercoated by 410SS between 316SS substrate and  $Y_2O_3$  coating with densification treatment (case 4) in the thermal shock test