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**COMPATIBILITY TEST OF BLANKET STRUCTURAL MATERIALS
WITH BERYLLIUM SPHERE IN HELIUM GAS ENVIRONMENT**

March 1995

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Compatibility of blanket structural materials with beryllium spheres under the helium environment was studied at temperatures of 650-750°C for periods of 700 and 1500hr. The examined materials were 316 stainless steel as a reference material and advanced structural materials such as titanium alloy (Ti-6Al-4V), reduced activity ferritic steel (F82H) and vanadium, all of which are candidate materials in the ITER blanket design. Reactions of Ti-6Al-4V alloy and F82H with Be sphere in helium gas containing impurities were occurred at the test of 750°C, 700hrs and 700°C, 1500hrs. It was not observed at the test of 700°C, 700hrs. This indicated that these blanket structural materials are less compatible with Be and helium containing impurities under the condition of high temperature over 700°C. In the case of vanadium, oxidation was observed at 750°C, 1500hrs in the helium environment. Clear grain boundary grooving was formed on the vanadium surface contacted with He. No obvious surface reaction layer was formed in the helium exposure test of 700°C, 1500hrs. This preliminary result shows that upper temperature limit of these advanced materials is around 700°C, though compatibility capabilities are nominally greater than that of 316 stainless steel as the reference material.

This report partly corresponds to the ITER 1993 Emergency Task (Task No. JB-BL-14)

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* Toshiba Corporation

Keywords : Blanket Structural Materials, Beryllium Sphere, Compatibility Test, ITER
Blanket Design, F82H Ferritic Steel, Ti-6AL-4V, Vanadium

ブランケット構造材とベリリウム球のヘリウム雰囲気下での両立性試験

日本原子力研究所那珂研究所核融合工学部

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小野 清^{*}・小林 重忠^{*}

(1995年2月1日受理)

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この報告はITER1993緊急タスク(Task No. JB-BL-14)の一部をまとめたものである。

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1. Introduction

In view of the fusion blanket design strategy, material evaluation should include different sets of structural materials/coolant /tritium breeding materials combinations. It is clear then that major materials issues could differ by materials, operating temperature ranges and service environments.

In the design of the ITER blanket, two options have been examined, a tritium breeding blanket and a non-breeding blanket. A layered pebble bed type breeding blanket with water cooling was proposed by JAERI during ITER /CDA and EDA. Another tritium breeding blanket is an advanced design option based on liquid lithium with vanadium alloy as a structural materials, because it offers significant advantages relative to stainless steel in terms of high heat flux accommodation. Regarding the structural materials, another option is applied titanium alloys instead of vanadium alloys. Titanium alloys (typically Ti-6Al-4V) has been widely utilized due to its low density, high ratio of mechanical strength to density. The qualification data of these advanced materials, vanadium and titanium alloys, are limited compared with stainless steel. Low activation characteristic is also an attractive feature in case of application to nuclear components such as in-vessel components of fusion reactors. A reduced activated ferritic steel (typically F82H) could be applied to the blanket material in future. This improved ferritic steel was developed by JAERI and Nippon Kokan K.K. High thermal conductivity and low thermal expansion of F82H steel are the advantages for use in the high heat flux environment.

These advanced materials, however, have a high affinity with beryllium (Be) and impurities in the flowing gas. Therefore, compatibility test with Be and helium gas is necessary to be conducted for evaluation of feasibility of these advanced structural materials.

2. Experimental description

The compatibility tests of blanket structural materials with Be in the helium environment were studied at temperatures of 650 - 750°C for periods of 700 and 1500 hrs. The blanket structural materials examined were vanadium, titanium alloy, ferritic steel and type 316 austenitic stainless steel (316SS), all of which were candidate materials in the ITER blanket design. Compatibility of these blanket structural materials with Be is known to be sensitive to the impurities in the helium gas. When they were exposed to even fairly low partial pressure of impurities in the helium

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gas, it may induce oxidation and embrittlement phenomena. Thus, chemical reactivity of these structural materials is one of the major concerns to apply this combination to the ITER blanket design.

The examined structure materials are titanium alloys (Ti-6Al-4V), reduced activity ferritic steel containing 8% chromium (F82H), vanadium and 316SS. As the ITER candidate vanadium alloys (V-5Cr-5Ti) is not available for the limited period, pure vanadium metal is used in this helium exposure test. The chemical contents of these blanket structural materials are described in Table 1. The specimens were constituted of disks (10-15 mm in diameter and around 1 mm thick). Contact faces of disks were polished with emery papers. A stack of beryllium sphere and blanket structural materials was set in a stainless steel capsule and a weight was put on them to insure a contact pressure of 34kPa. The tests were carried out under flowing helium (99.99% purity) with the flow rate of 1.8 l/h. Impurities in the helium gas were water, oxygen, nitrogen and hydrogen etc. and each amount is about 20 vppm, respectively. The helium gas flew through the stack along the grooves of specimens. The capsules were heated by an electrical furnace and isothermally annealed at 650-750°C for 700 and 1500 hrs. Figures 1 and 2 show diagrams of the compatibility test apparatus and sample setup, respectively.

After annealing, the stack of specimens was fixed with a resin and small piece containing all of the contact surfaces between the stacked specimens was obtained. The piece was then cut longitudinally and the cross sections of the two halves were polished with emery papers up to 1200 grade. To reveal the aspect of the reaction zones clearly, the specimens were finally treated with electrolytic etching in a solution (mixture of 100 ml of 36% hydro-chloric acid, 10 ml of 61% nitric acid, and 100ml of distilled water) at 30°C around for 5 second. The specimens were examined with a scanning electron microscope (SEM). The depths of the reaction zone were determined from the SEM micrographs. The element distributions were analyzed by electron dispersion X-ray analysis (EDX) and electron scattering chemical analysis (ESCA).

3. Experimental results

This experimental objectives are to obtain engineering database on compatibility of blanket structural materials. As test period is not enough long, these experiments give a preliminary result on corrosion rate of Ti alloy, ferritic F82H steel, vanadium and 316SS by flowing helium gas

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containing small amount of impurities (below 20vppm, oxygen, water and nitrogen, respectively).

Figures 3 -5 show the typical surface aspects of the blanket structural materials and Be spheres before compatibility test. The SEM micrographs were obtained with the specimens prepared with electrolytic etching .

3.1 Titanium alloys

Experimental results of the contact tests are summarized in Table 2. This indicates that titanium alloy (Ti-6Al-4V) is less compatible with Be and helium containing impurities under the condition of high temperature over 700°C. This result shows that reaction of Ti-6Al-4V alloy with Be sphere initiates at around 700°C.

Structures of the reaction dent in Ti alloy were developed on the contact surface after 1500 hrs tests at 700°C. Figure 6 shows the SEM micrograph of the structure of the reaction dent. This bird's-eye view shows that reaction initiates from the original surface of the steel and forwarded into the Be side and made dent after removing Be with chemical etching. On the other hand, same structure was not observed at the test of 700°C, 700hrs as shown in Fig. 7. The element analysis by EDX was conducted to the test samples of 750°C,1500 hrs as indicated in the Fig. 8. No segregation of major constitutional element was observed. Figure 9 shows ESCA spectrum to relate the existence of the oxygen. This was also indicated surface reaction by oxidation in the He environment containing small water and oxygen.

3.2 Ferritic steel (F82H)

Experimental results of the reaction tests are summarized in Table 2. In this table, reaction of F82H with impurities containing helium were occurred at 750°C, 700 hrs and 700°C, 1500 hrs. It was not occurred at 700°C, 700 hrs. Figure 10 shows typical aspects of the Be-steel interaction. As shown in this figure, reaction was not occurred at 650°C, 1500 hrs. This indicates that F82H steel is less compatible with Be and helium containing impurities under the condition of high temperature over 700°C. Figure 11 is a result of EDX analysis which indicates no segregation of Fe and Cr in the bulk matrix. Compatibility capability of F82H steel is nominally better than that of 316SS [1] as shown in the Table 2.

3.3 Vanadium

In the case of vanadium, test was conducted in the condition of without Be sphere. Oxidation of vanadium was observed at 750°C, 1500 hrs in the helium environment. As shown in Fig. 12, clear grain boundary grooving was formed on the vanadium surface contacted with He. And oxygen peak by ESCA analysis (Fig. 13) was observed, indicating existence of oxide layer on the surface of vanadium. No obvious surface reaction layer was formed in the lower temperature test of 700°C, 1500 hrs in Fig. 14.

3.4 316 stainless steel

The SEM micrographs of 316SS indicates typical aspects of the beryllium-steel interaction as the double layer structure. The double layer structure of the reaction zone was developed on the contact surface after 700 and 1500 hrs tests of 650, 700 and 750°C. Figures 15-18 show that the inner layer of the double layered reaction zone initiates from the interface on the original surface of the steel. On the other hand, outer layer proceeds to the original beryllium and consists of intermetallic compounds. Total thickness of the reaction zone in contact with Be sphere is coincident with the case of compatibility with plate contact beryllium. Figure 19 is a result of EDX analysis which indicates no segregation of Fe and Cr in the bulk matrix. Compatibility capability of 316SS seems to be worse than that of F82 ferritic steel as shown in the Table 2.

4. Conclusions

The compatibility tests of the blanket structural materials with Be under helium environment were conducted at temperatures of 650 - 750°C for periods of 700 and 1500 hrs. The examined structural materials were type of titanium alloy, ferritic F82H steel, vanadium and 316SS, all of which were the candidate materials in the ITER blanket design. Following conclusions were obtained from this experiment.

Reaction zone of Ti alloy and F82H were observed at 750°C, 700 hrs and was not observed at 700°C, 700 hrs. But it was observed at test of 700°C, 1500 hrs. This indicated that these advanced materials are less compatible with Be and helium containing impurities under the condition of high temperature over 700°C. In the case of vanadium, oxidation was observed at 750°C, 1500 hrs in the helium environment. Clear grain boundary grooving is formed on the vanadium surface contacted with He. No obvious surface reaction layer was formed in the helium exposure test

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The double layer structure of the reaction zone of 316 stainless steel was developed on the contact surface after 700 and 1500 h tests of 650, 700 and 750°C. The inner layer of the double layered reaction zone was formed on the contact surface with nearly constant depth. On the other hand, outer layer proceeds to the original Be and consists of intermetallic compounds. Total thickness of the reaction zone in contact with Be sphere is coincident with the case of compatibility with plate geometry Be.

Further works are required to investigate the feasibility and applicability of these blanket materials in the fusion experimental and demo reactors.

Acknowledgement

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Fusion Technology 1992, P1547, Proceedings of the 17th
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Table 1 Chemical contents of advanced structural materials

Titanium alloy (Ti-6Al-4V)

Content	Ti	C	V	Al	N	O	Fe	H
Wt%	Bal	0.01	4.11	6.37	0.01	0.17	0.13	0.00009

Ferritic Steel (F82H)

Content	C	Si	Mn	P	S	Ni	Cr	Mo	W
Wt%	0.097	0.09	0.07	0.002	0.003	0.03	7.46	0.001	2.1

"	V	Nb	Ta	Co	Ti	B	Al	N	O
"	0.18	0.00007	0.03	0.005	0.008	0.0004	0.014	0.004	0.003

Vanadium

Content	Al	Co	Fe	Mo	Ni	Si
Wt%	0.01	0.001	0.006	0.01	0.001	0.01

316 stainless steel

Content	Cr	Ni	Mo	Mn	Si	C	P	S
Wt%	16.3	11.2	2.07	1.16	0.29	0.05	0.04	0.005

Table 2 Reaction thickness of advanced material with Be/He

Test No	Temp. (C)	Time (Hour)	Reaction thickness (μ m)				
			316 SS		F82H		Ti ₆ Al ₄ V
			NGK	B.W	NGK	B.W	
Test1	750	1500	72.2	86.1	-	5.6	30.6
Test2	700	1500	72.2	13.9	16.7	30.6	44.4
Test3	650	1500	44.4	16.7	0	0	0
Test4	750	700	61.1	83.3	-	27.8	19.4
Test5	700	700	55.6	44.4	-	0	0
Test6	650	700	19.4	19.4	0	0	0

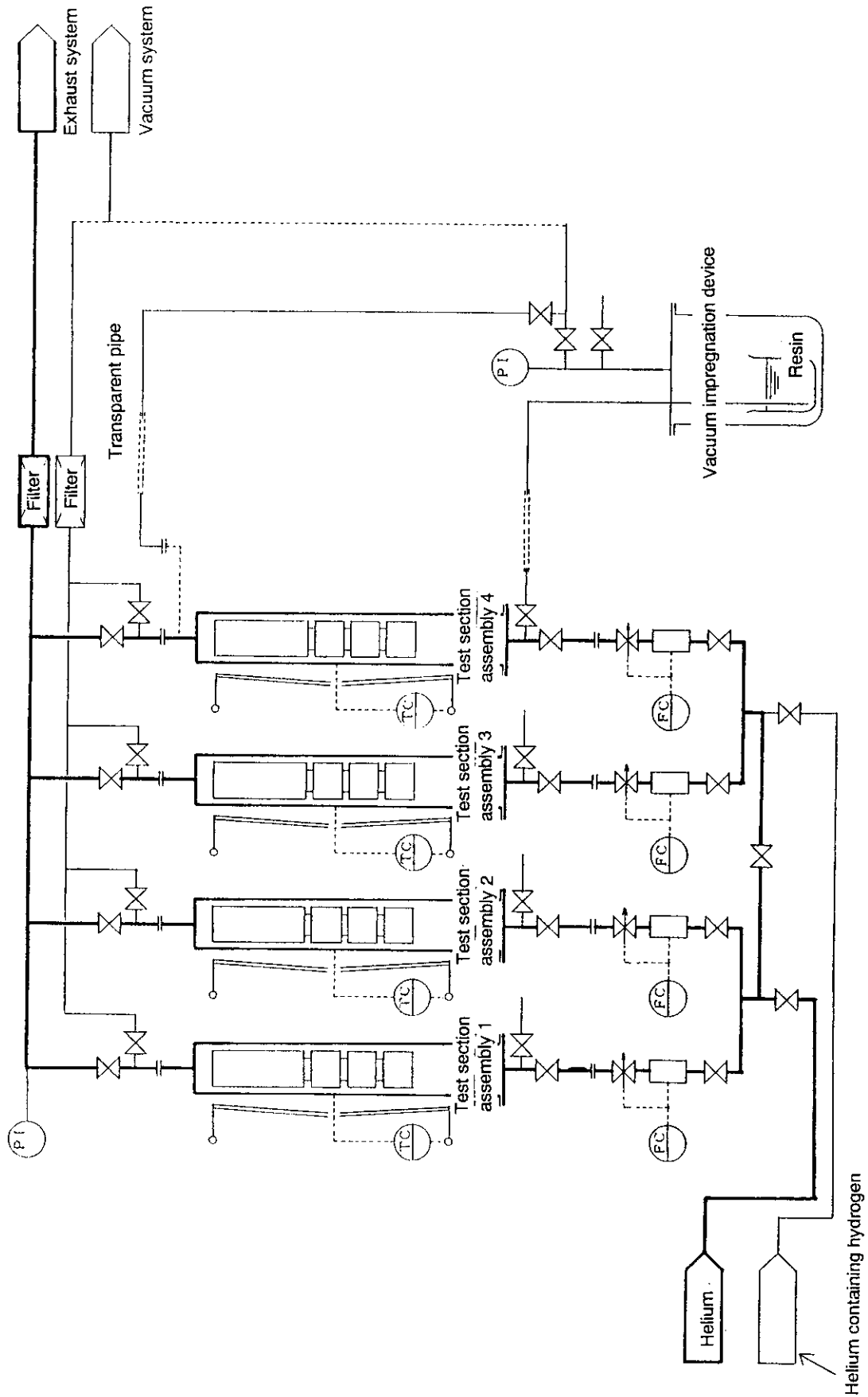


Fig. 1 A compatibility test apparatus.

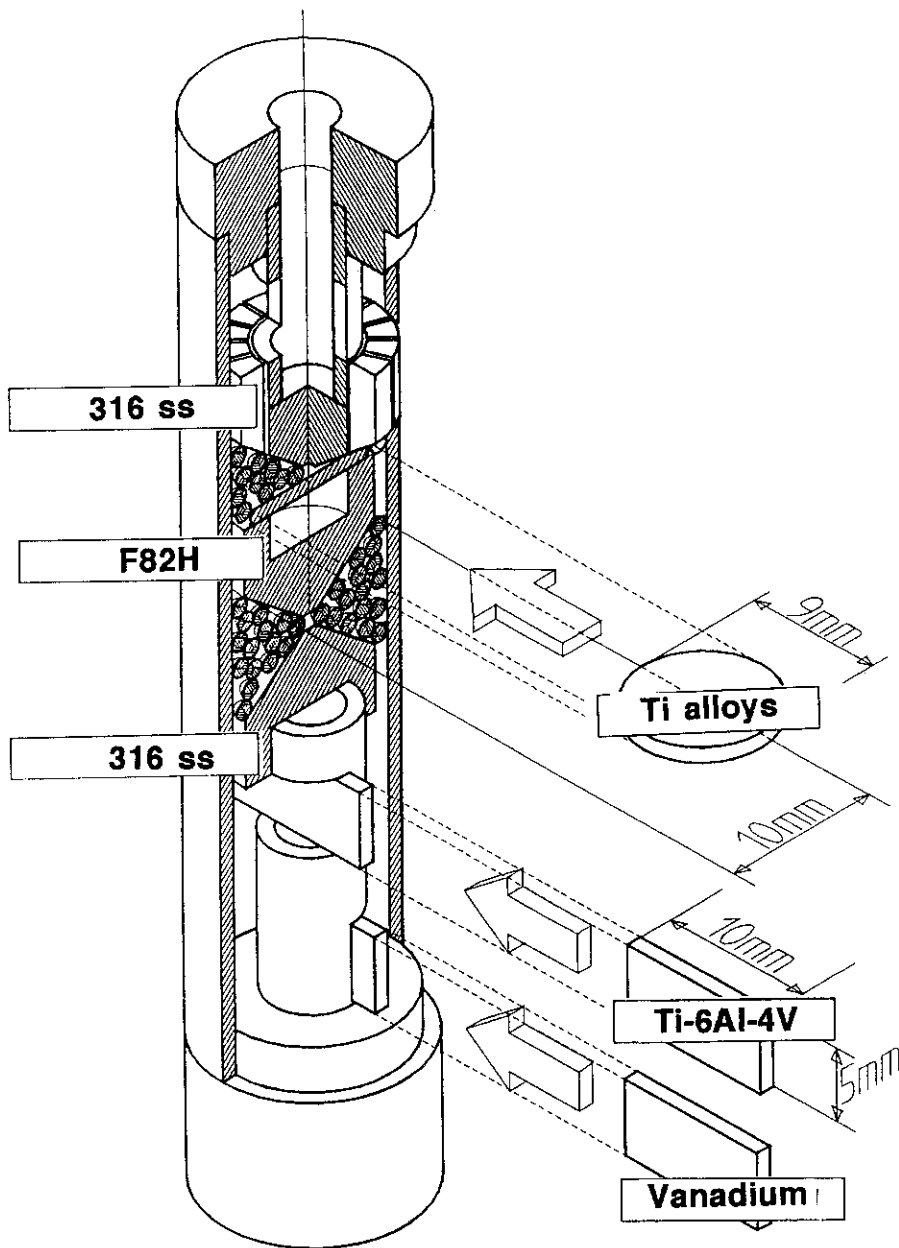


Fig. 2 Diagram of sample setup

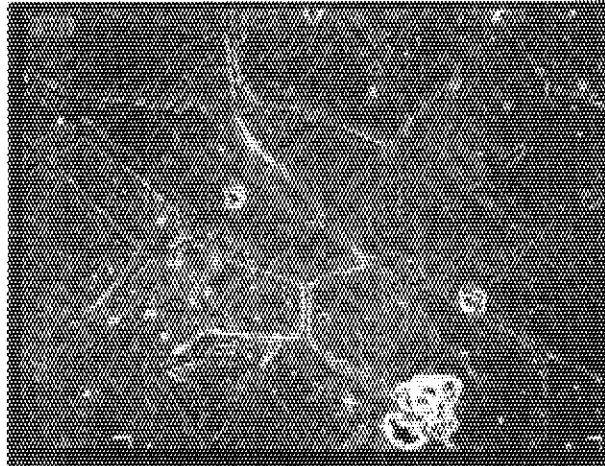


Fig. 3 A SEM photograph of material surface before test (Vanadium)
($\times 500$)



Fig. 4 A SEM photograph of material surface before test (F82H)
($\times 500$)

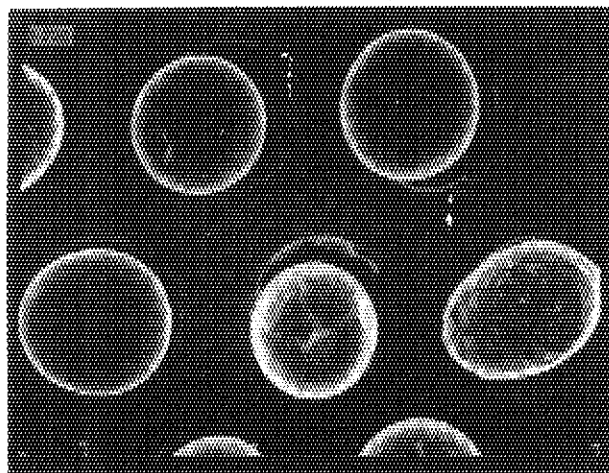


Fig. 5 A SEM photograph of Be sphere before test
(cross section view : $\times 30$)

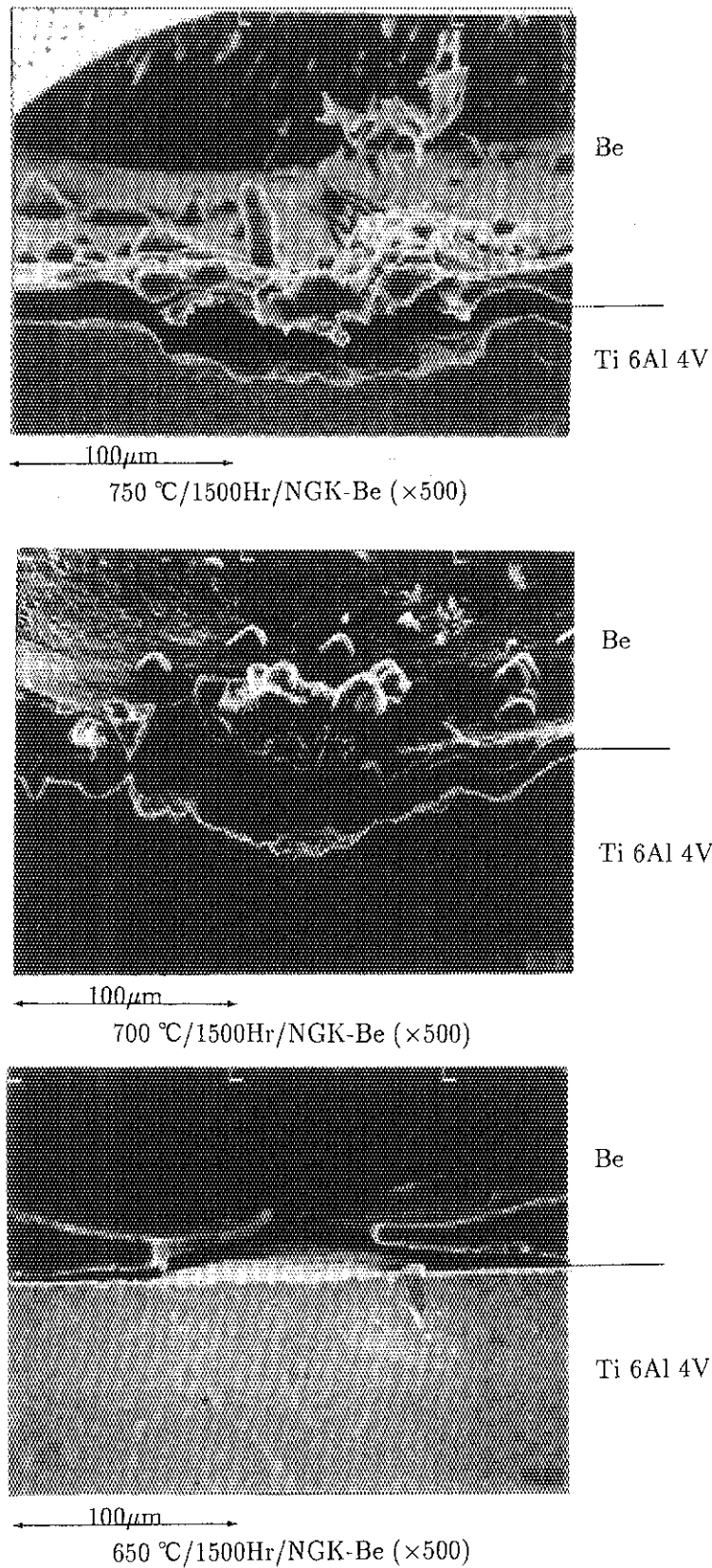


Fig. 6 A SEM photograph of cross section view of titanium alloy after test.
Up : 750°C/1500Hr/Be
Middle : 700°C/1500Hr/Be
Bottom : 650°C/1500Hr/Be

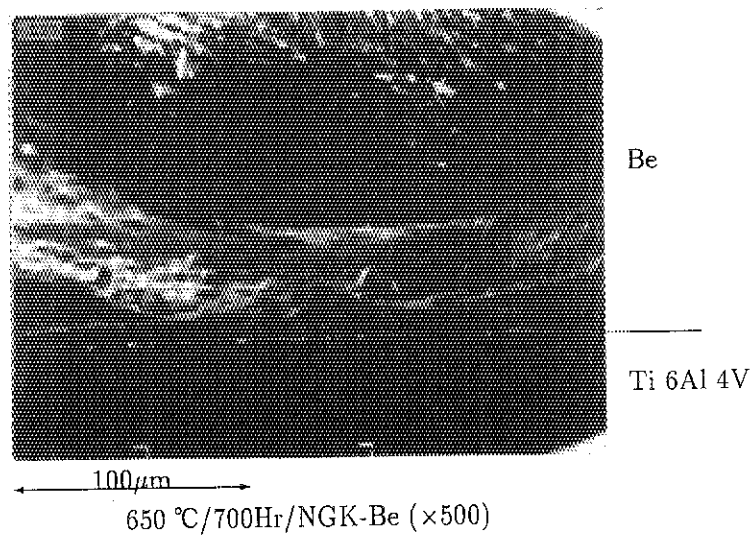
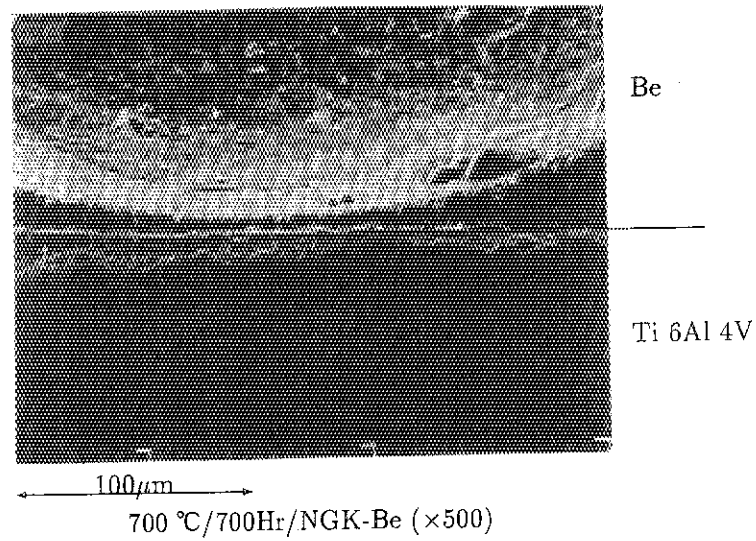


Fig. 7 A SEM photograph of cross section view of titanium alloy after test,
Up : 700°C/700Hr/Be, Bottom : 650°C/700Hr/Be

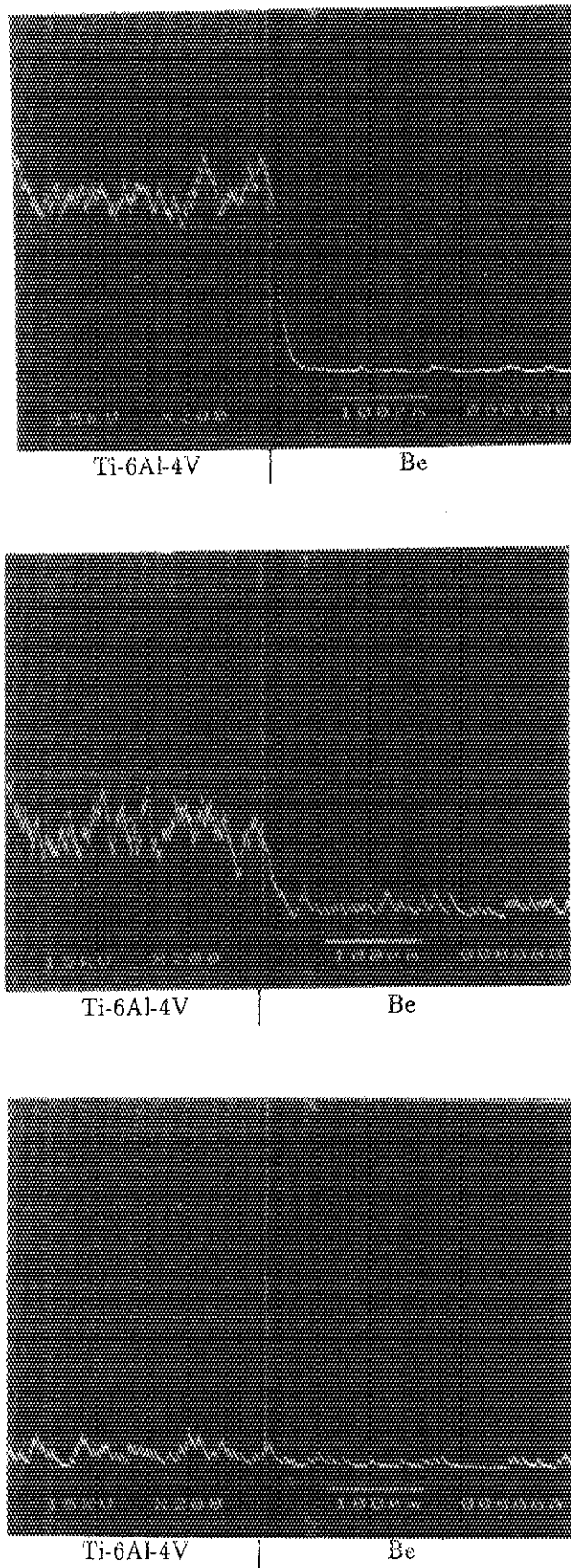


Fig. 8 EDX line analysis of titanium alloy (750°C/1500Hr/Be)
Up : Ti, Middle : Al, Bottom : V

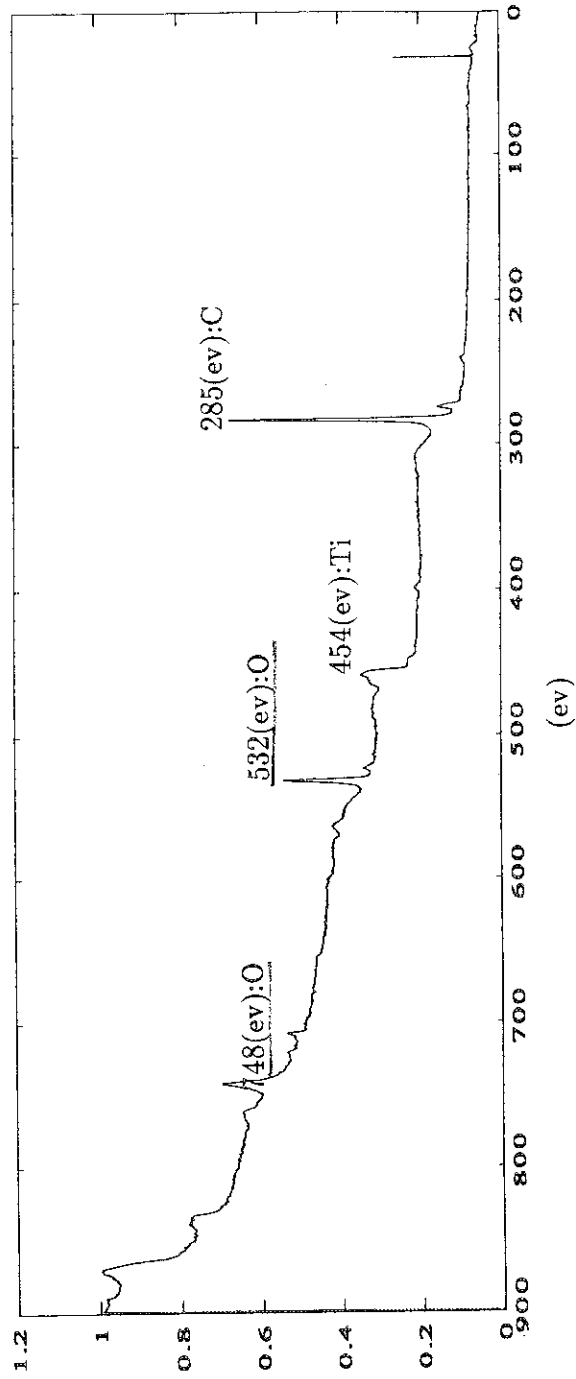


Fig. 9 ESCA spectrum of titanium alloy after test
(750°C/1500Hr/Be)

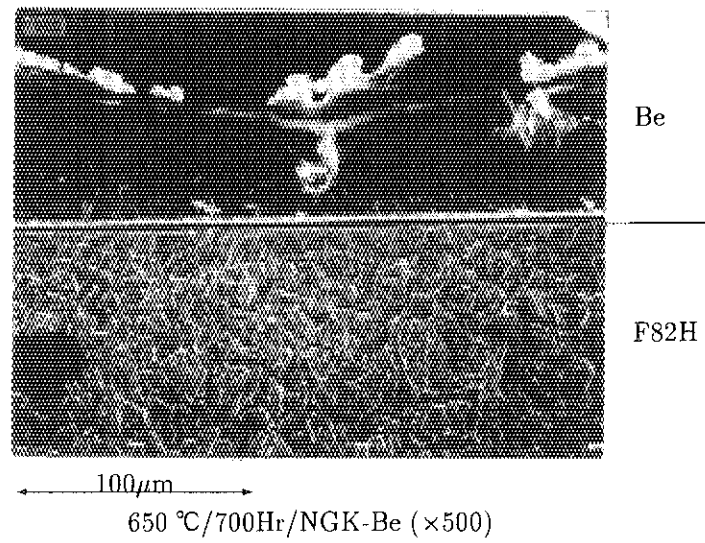
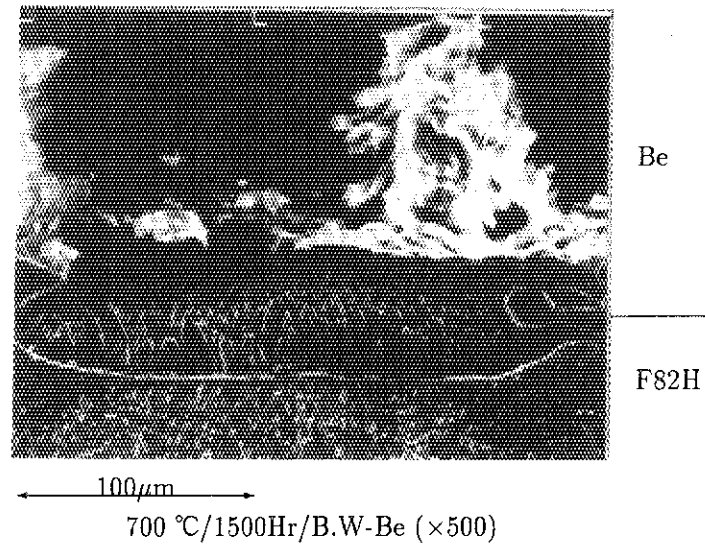


Fig.10 A SEM photograph of cross section view of F82H after test
Up : 700°C/700Hr/Be, Bottom : 650°C/700Hr/Be

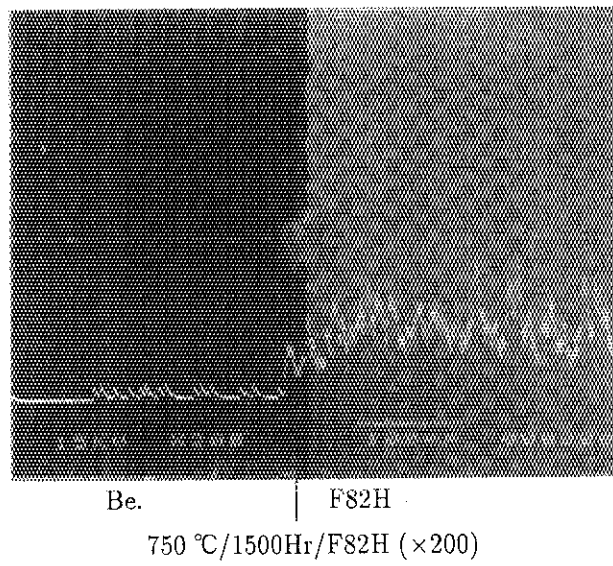
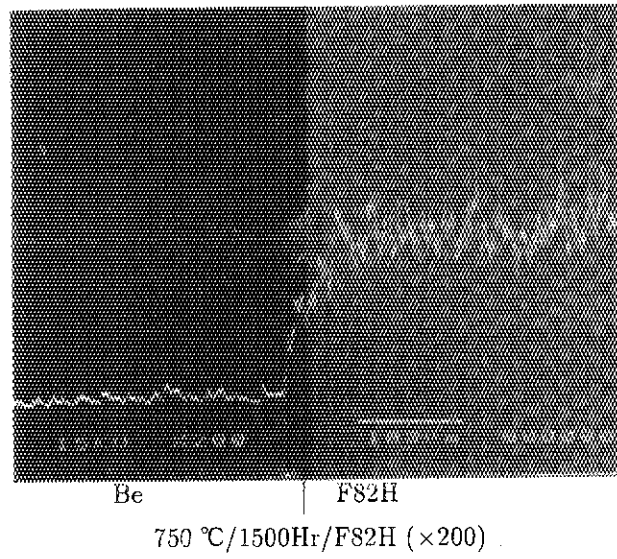


Fig.11 EDX line analysis of F82H after test (750°C/1500Hr/Be)
Up : Fe, Bottom : Cr

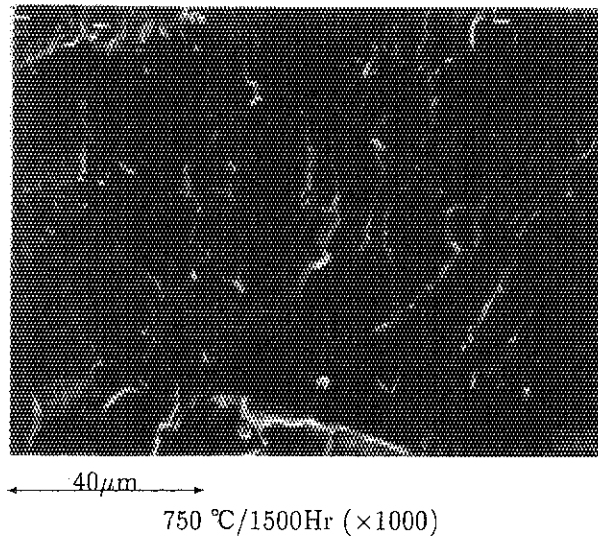


Fig. 12 A SEM photograph of Vanadium after test of He environment

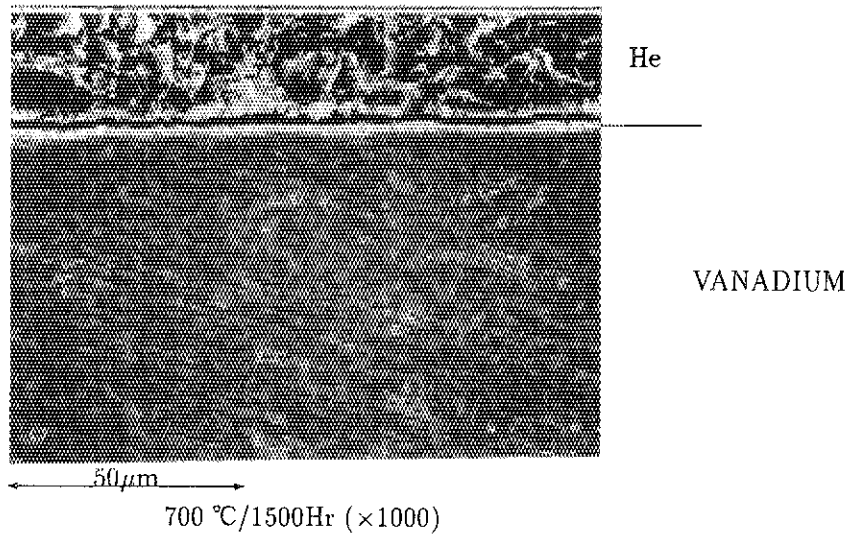


Fig. 14 A SEM photograph of material surface after test
(Vanadium : 700°C 1500Hr, ×1000)

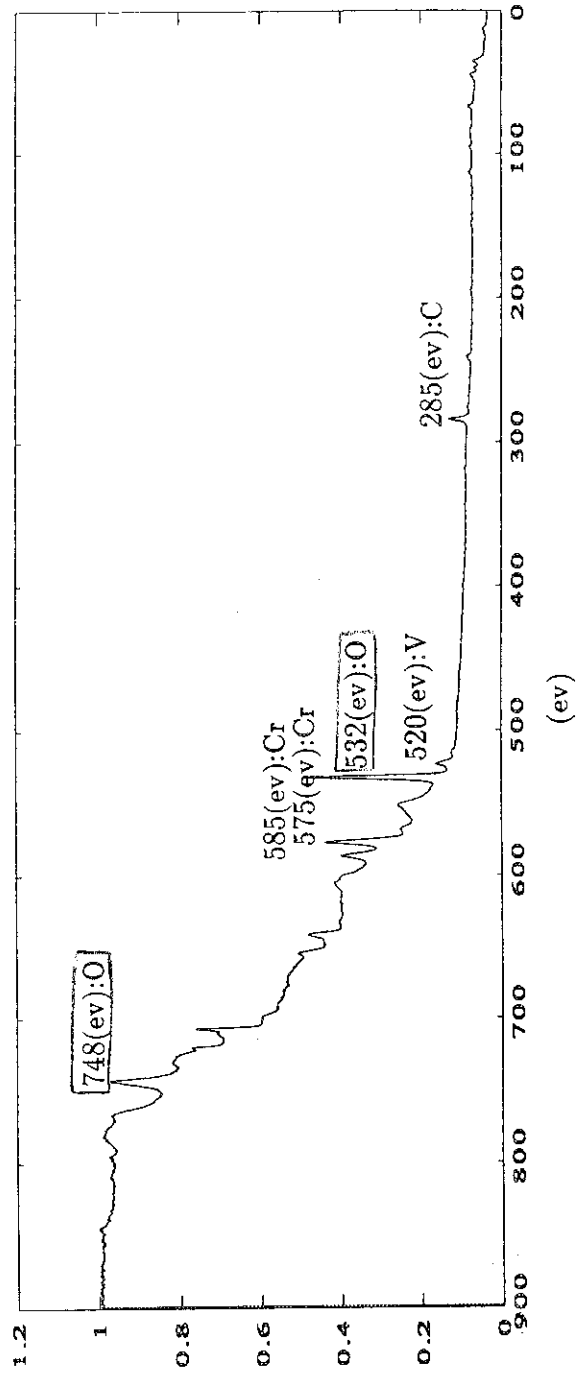


Fig.13 ESCA spectrum of vanadium after test
(750°C/1500Hr/Be)

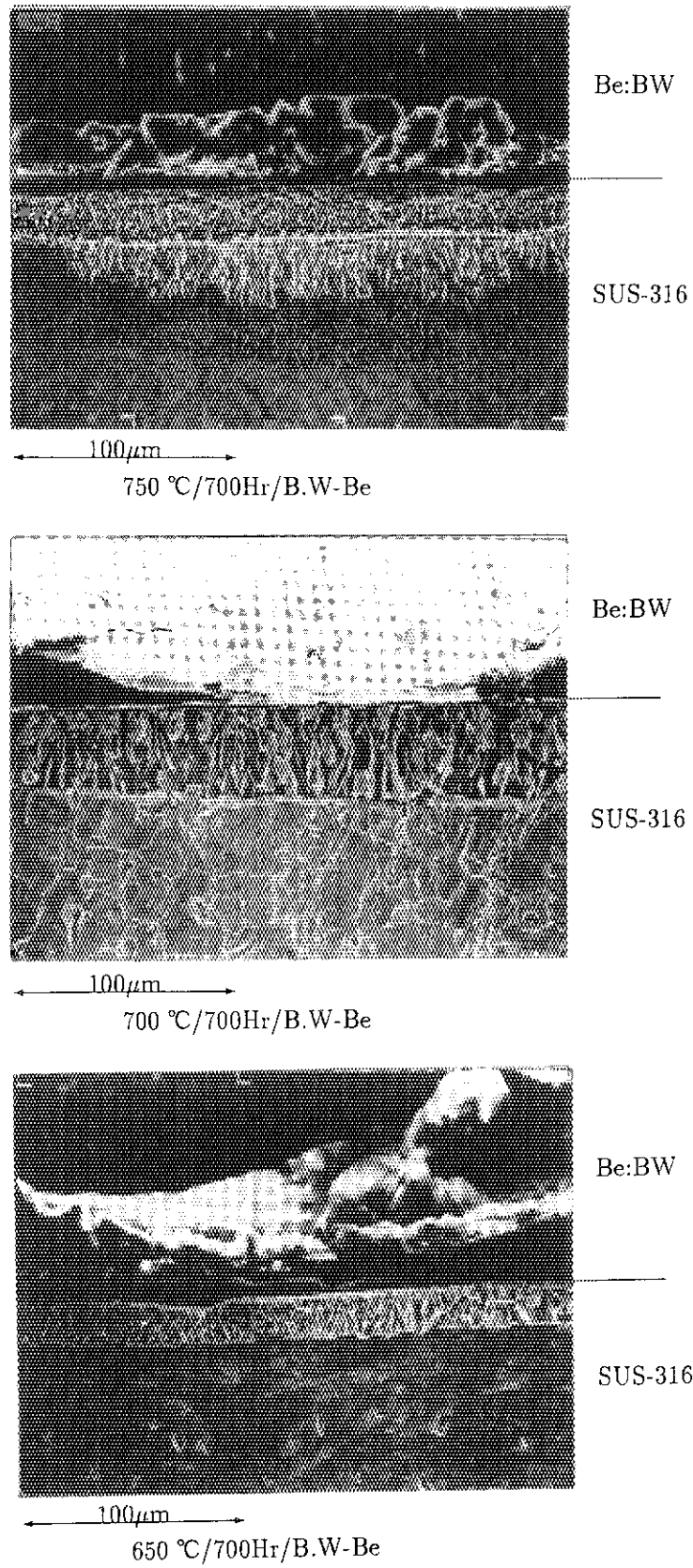


Fig. 15 A SEM photograph of cross section view of 316SS after test used NGK-Be sphere
Up ; 750°C/1500Hr, Middle ; 700°C/1500Hr, Bottom ; 650°C/1500Hr

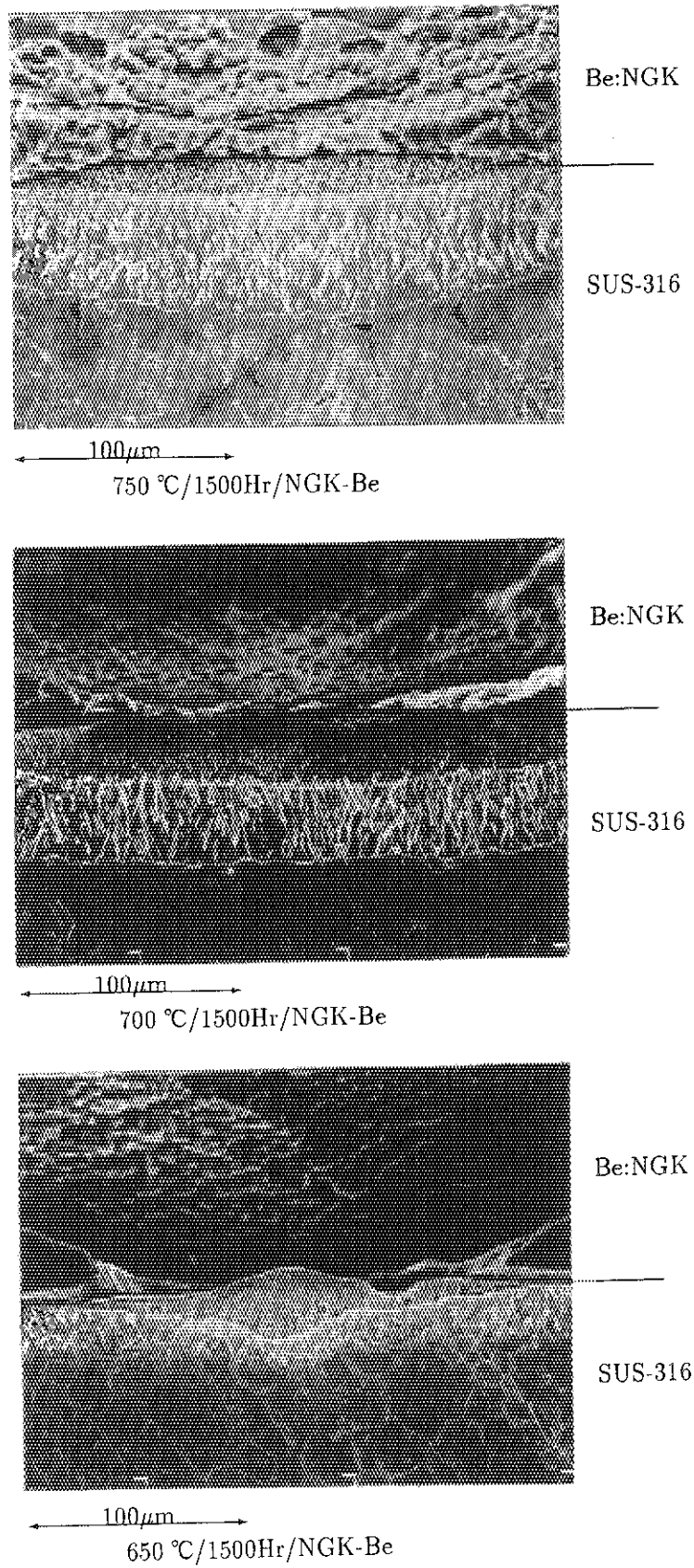


Fig. 16 A SEM photograph of cross section view of 316SS after test used Brush wellman-Be sphere
Up ; 750°C/1500Hr, Middle ; 700°C/1500Hr, Bottom ; 650°C/1500Hr

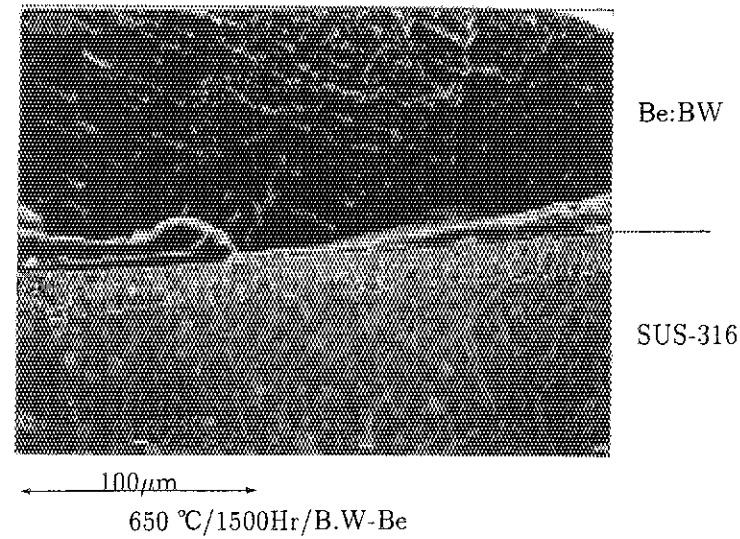
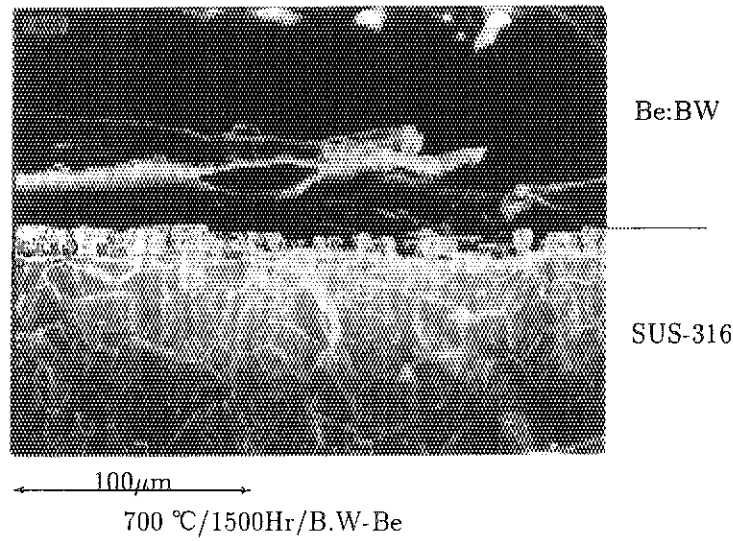
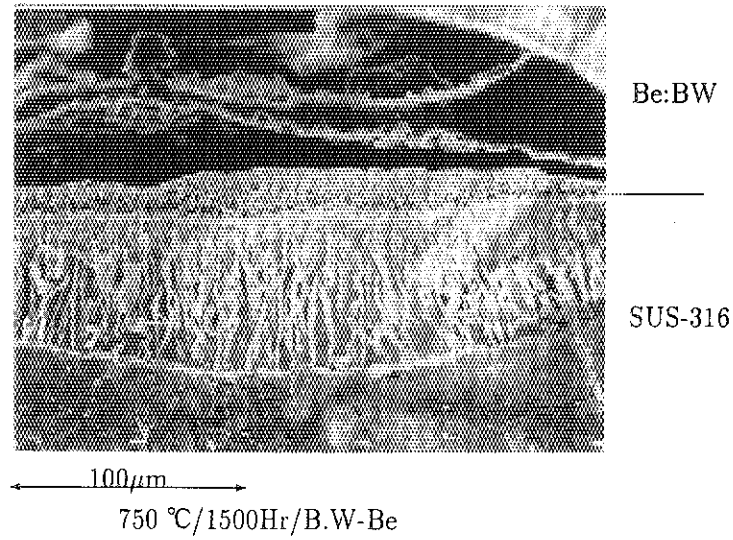


Fig. 17 A SEM photograph of cross section view of 316SS after test used NGK-sphere
Up ; 750°C/700Hr, Middle ; 700°C/700Hr, Bottom ; 650°C/700Hr

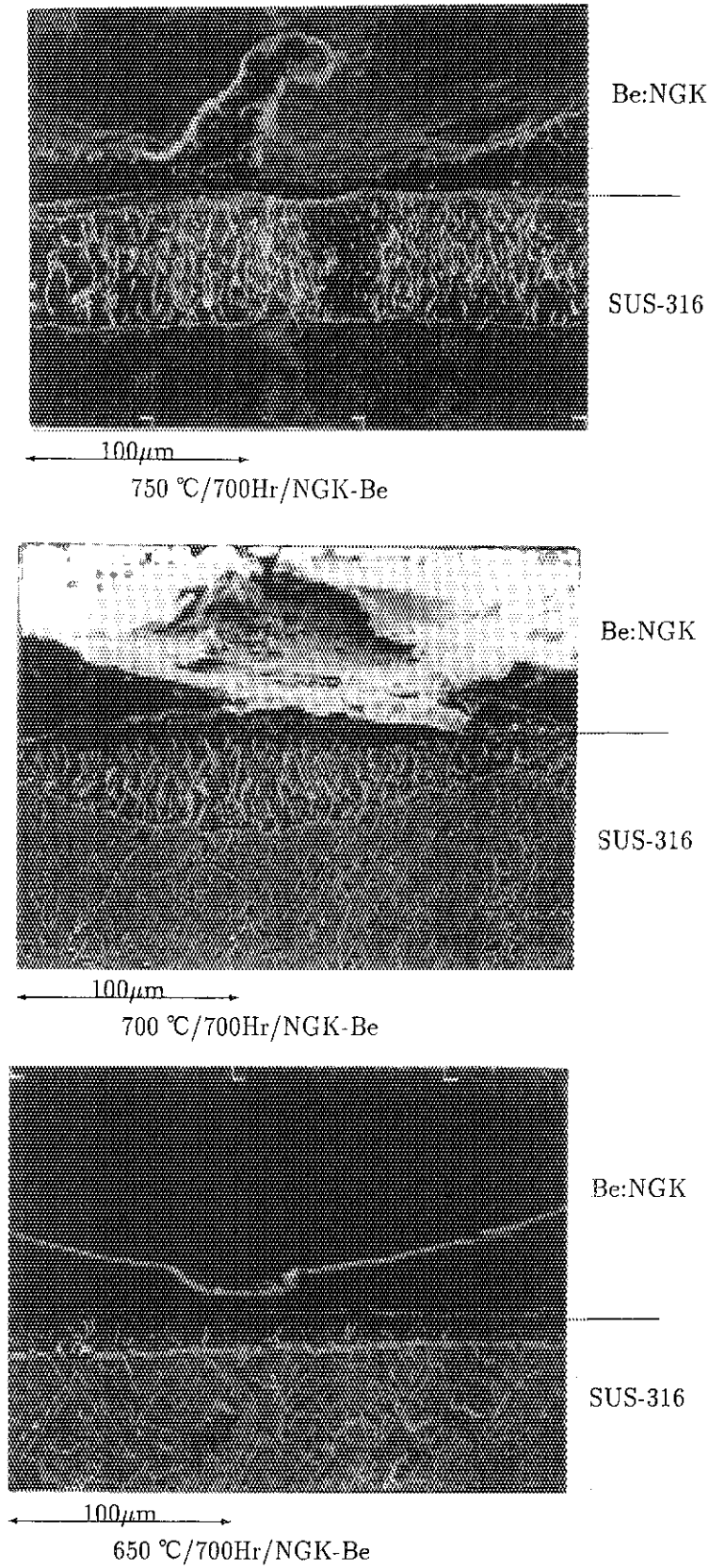


Fig. 18 A SEM photograph of cross section view of 316SS after test used Brush wellman-sphere,
Up ; 750°C/700Hr, Middle ; 700°C/700Hr, Bottom ; 650°C/700Hr

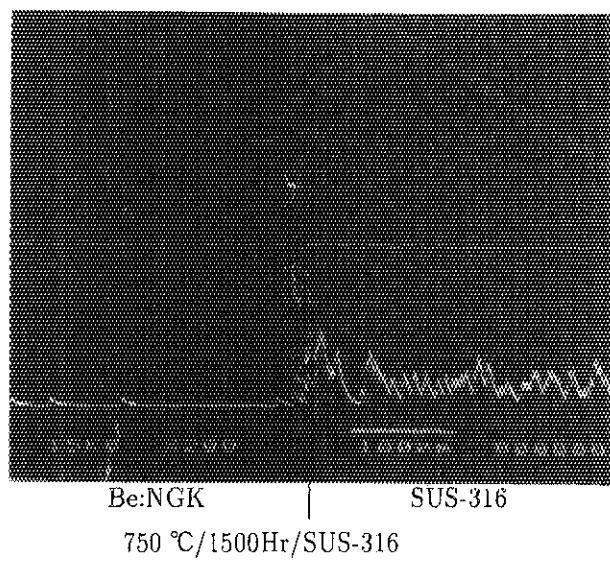
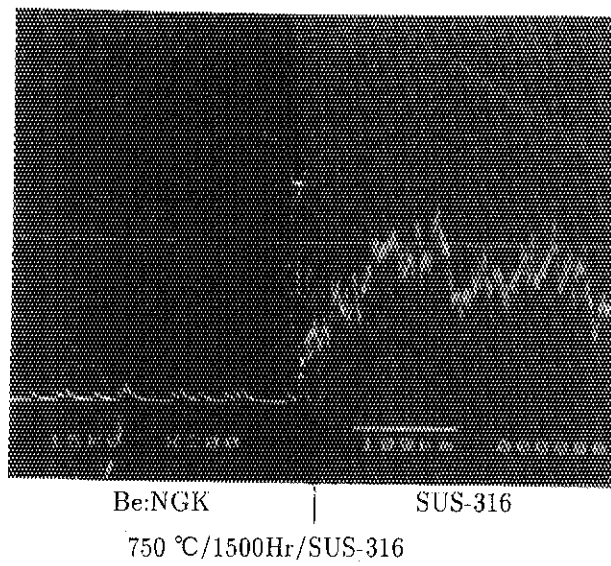
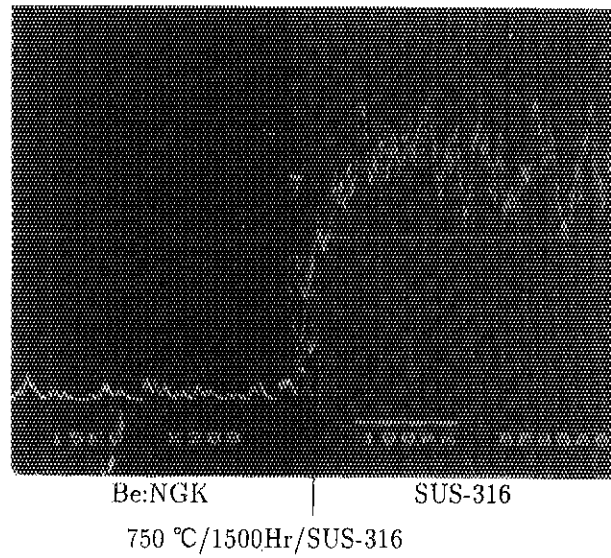


Fig. 19 EDX line analysis of 316SS after test of 750°C, 1500Hr.
Up ; Fe, Middle ; Cr, Bottom ; Ni