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(Received February 18, 1986)

In studying the carburization behaviour of high temperature alloys, carburization tests in heavy carburizing environment were undertaken on several potential candidate alloys for uses in HTR system. The alloys tested were R 4286 developed by ERANS (Engineering Research Association of Nuclear Steel-making) programme, Ni-18.5 % Cr-21.5 % W as identified F alloy, Hastelloy XR and Hastelloy XR-II, and the test environment conditions employed were (Ar + 10 % CH_4) gas at 800, 850, 900 and 950 °C for up to 100 hours.

It is shown that when no protective surface oxide layer is formed, the carburization kinetics follows a parabolic rate law, and the alloying additions of Al and Ti have a beneficial effect in dry environment (very low oxidizing) on the carburization resistance due to the formation of Al-Ti-based oxide and without formation of Cr-based oxide. In the comparison of the carburization resistance of the alloys tested, R 4286 containing 2 % Al and 2.5 % Ti exhibits the best resistance due to the formation of the protective Al-Ti-based oxide and the F alloy shows a moderate resistance, while Hastelloys XR and XR-II are found to have the lowest resistance.

Keywords: High Temperature Alloys, ERANS Alloys, R 4286, Ni-Cr-W Alloy, Hastelloy XR, Hastelloy XR-II, Carburization, HTR

浸炭雰囲気中の耐熱合金の浸炭挙動

日本原子力研究所東海研究所燃料工学部
新藤雅美

（1986年2月18日受理）

高温ガス炉用耐熱合金の浸炭挙動を調べるために，強浸炭雰囲気中での浸炭試験を行った。試験に用いた材料は，ERANS（Engineering Kesearch Association of Nuclear Steel－making －原子力製鉄技術研究組合－）合金の一つであるR 4286，Ni－18.5％Cr－21.5％W（F合金），ハステロイXR及びハステロイXR－Ⅱである。試験は800℃，850℃，900℃及び950℃の（Ar＋10％CH_4）雰囲気中で100ｈまで行い，次のような結果が得られた。

保護酸化膜が形成されない場合，浸炭速度は放物線則に従う。Ｃｒの酸化膜が形成されない様な低酸化ポテンシャル環境では，Aℓ及びＴｉの添加が，それらの保護膜の形成によって，耐浸炭性を改良する。耐浸炭性を合金間で比較すると，2％のAℓと2.5％のＴｉを含んだR4286がAℓ及びＴｉからなる保護膜によって，もっとも優れた特性を示した。また，Ｆ合金は中間の，ハステロイＸＲ及びＸＲ－Ⅱはもっとも低い耐浸炭性を示した。

東海研究所：茨城県那珂郡東海村白方字白根２の４

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1. INTRODUCTION

A lot of materials tests, on the items such as creep, fatigue and corrosion , of high temperature alloys have been carried out in the helium-based test environments in various laboratories for the qualification of those materials in application to the process heat high temperature reactor (HTR).

Among the effects of the gas-metal interactions expected in the HTR environment, carburization and/or decarburization is supposed to have strong influence on the mechanical properties. For the metallic materials such as Hastelloy alloys X and Inconel 617, carburization in most standardized simulated HTR environments, like JAERI type B and/or German Prototypanlage Nukleare Prozesswaerme (PNP) helium do not cause serious carburization problem[1,2] except in crevices, where formation of stable Cr-based oxide scale is hardly attained due to local depletion of oxidizing species[3].

It is, however, uncertain whether the similar is always the case in the actual environment in process heat HTRs with outlet temperature above about 850°C, where the chemical reaction rate is very high.

As for the environmental effect on mechanical properties, it has been reported that effect of carburization on creep design parameters, such as rupture time and time to 1% strain, has not been observed[2], while heavy carburization causes significant loss of ductility in room temperature tests[4]. On the other hand, decarburization has a substantial effect on the creep behaviour. Decarburized specimens generally exhibit decreased creep strength[5].

In West Germany and the U.S.A. carburization which is one of the environmental effects has been considered seriously, and most of the developmental alloys have chemical composition with comparatively low Cr and high Ti and Al contents for increased carburization resistance[6,7,8].

It is quite possible that the real reactor atmosphere, especially during normal operation, will be carburizing environment. Furthermore, if a service environment is too dry in which Cr-based oxide can not be formed, carburization will be a serious problem as mentioned above.

The aim of the work reported here is to investigate the carburization behaviour of two heats of Hastelloy alloys XR, Ni-Cr-W alloy and one of the alloys developed by the Engineering Research Association of Nuclear Steel-making (ERANS). In the testing alloys, ERANS alloy which includes Al and Ti is given a focus because this alloy is expected to show a good carburization behaviour due to its potential of formation of Al and Ti based-oxide even in extremely dry environment.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The chemical compositions of the alloys tested are summarized in Table 1. The specimens were machined from the solution treated materials. The alloy R 4286, which was one of the alloys developed by ERANS, is strengthened mainly by γ' ($Ni_3(Al,Ti)$) precipitation, and is known to exhibit good carburization resistance in dry PNP environment due to the formation of Al(Ti) -based oxide scale[9]. The Ni-Cr-W alloy which is one of the second generation alloys is strengthened by α-W precipitation, while Hastelloy

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XR-II is a high creep strength version of Hastelloy XR due to the additions of 40 ppm B.

Specimens for carburization test are machined in the type of hemidiscoid of 13 mm diameter x 2 mm thickness for R 4286 and Ni-Cr-W (F) alloy, while the specimens of the type of coupon of 5 mm x 10 mm x 2 mm were used for Hastelly alloys XR. Prior to the test, the surface of specimens was finished by wet abrasion paper (# 220).

2.2 Test Conditions

Carburization tests were carried out at 800, 850, 900 and 950°C for up to 100 hours in (Ar + 10% CH_4) gas. The impurities,such as H_2O and O_2, in the gas were not measured.

The specimens exposed were evaluated by change in carbon content and metallography. For the determination of the change in carbon content averaged over the cross-section, bulk carbon analysis of the specimens after removing some surface products by wire brush was undertaken.

3. EXPERIMENTAL RESULTS

3.1 Change in Carbon Content

The carbon uptake curves of Hastelloy XR, Hastelloy XR-II, F alloy and R 4286 at tested temperatures are summarized in Fig.s 1, 2, 3 and 4, respectively. Figure 1 shows the results of Hastelloy XR-II which indicates that the carbon uptake caused by carburization increased with

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raising temperature and the increase in carbon content with time showed a more or less parabolic relationship except the results at 800°C which probably followed a linear rate law. The results of Hastelloy XR shown in Fig.2 are mostly comparable to those of Hastelloy XR-II in Fig.1.
From Fig.3 which is the carbon uptake curves of F alloy, it can be seen that the increase in carbon content at 800 and 850°C followed a linear rate law, while those at 900 and 950°C followed a parabolic rate law.
For convenience, the carburization rates for R 4286 at 900 and 950°C were assumed to follow a parabolic rate law, although the carbon uptake curves shown in Fig.4 were not so systematic.

For the determination of the carburization kinetics of each alloys, the increase in cabon content must be expressed as mass of carbon increase[10] per unit surface area, mg/cm^2, which was given by the following formula, not just as carbon content, ΔC (%) ;

$$\Delta M = \Delta C(W/A)/100$$

where ΔM = mass of carbon increase (mg/cm^2)

ΔC = increase in carbon content (%)

W = weight of untested specimen (mg)

A = surface area of the specimen (cm^2)

The comparative data for each alloys at several temperatures are summarized in Fig.s 5, 6 and 7. From these figures, it can be found with respect to carburization resistance that R 4286 was the best one, F alloy exhibited moderate property and Hastelloy alloys XR showed the lowest resistance in the alloys tested. Concerning the carburization resistance of Hastelloy alloys XR , it has been reported[11] that addition of B in Hastelloy XR caused higher carbon uptake rate in JAERI type B helium. However, contrary to the expectation, i.e. the carbon uptake of Hastelloy XR-II containing 40 ppm B was higher than that of Hastelloy XR, no influence of B addtion was observed in heavy carburizing environment.

From Fig.s 5, 6 and 7 (results of R 4286 and F alloy at 850°C were assumed to follow a parabolic rate law) except the results at 800°C, parabolic rate constants were calculated venturously, although some data points deviated from a parabolic rate law. The temperature dependence of carburization rate assumed to follow parabolic rate law is shown in Fig.8. As can be seen from this figure, the logarithm of parabolic rate constants except the data points of R 4286 is a linear function of the reciprocal of absolute temperature roughly and activation energies for Hastelloy alloys XR and F alloy are 387 and 467 KJ/mol, respectively.

3.2 Analysis of Surface Product

For the identification of the surface products formed on the alloys during exposure in the test environment, energy dispersive X-ray analysis was undertaken. The results were as follows ;

- for Hastelloy alloys XR, at 800°C surface products were Cr-Mn-based oxide, at 850, 900 and 950°C those were Cr-based carbide.
- for F alloy, at 800°C surface product was Cr-Mn-based oxide, at 850°C and 900°C those were Cr-based carbide, and at 950°C there was no surface product.
- for R 4286, at 800°C surface product was Cr-based oxide, at 850°C that was Cr-based carbide, at 900°C that was mixed product of Cr-based carbide and Al-Ti-based oxide, at 950°C surface of alloy was covered by Al-Ti-based oxide.

A typical example of the results is shown in Fig.9. In conclusion, Cr can not oxidize above 850°C in the test emvironment.

3.3 Microstructure of Carburized Specimen

Carburized structures of exposed specimens were observed even without etching, especially those at high temperatures like 900 and 950°C were remarkably detected. A typical example of carburized structures after exposure at 950°C is shown in Fig.10. It can be also made clear from this figure that the depth of carburized zone of R 4286 was shallower than those of the other alloys, which corresponded to a good carburization resistance of R 4286.

The microstructures of the specimens exposed at 800, 850, 900 and 950°C for each hours are shown in Fig.s 11, 12, 13 and 14, respectively. In most of carburized specimens, the carbide denuded zone was observed at the region near surface of alloy matrix. Unfortunately, the mechanism and process for the formation have not been become clear yet. From Fig.s 11, 12, 13 and 14, the depth of carburized zone was measured for the determination of carburization kinetics. The results measured are summarized in Fig.s 15, 16 and 17. With respect to the carburization resistance of F alloy, these results at 900 and 950°C are a bit different from the results of bulk carbon analysis. As described in section 3.1, according to the results of bulk carbon analysis, F alloy had a moderate carburization resistance. However, the carburization resistance referred to the carburized zone was nearly comparable to those of Hastelloy alloys XR. The reasons for the difference may be due to the precipitation process and/or the solubility limit of carbon in F alloy matrix, i.e. the precipitation of carbide and the solubility limit of carbon are faster and lower than those of Hastelloy alloys XR.

The temperature dependence of parabolic rate constants for the depth of carburized zone except the results at 800°C is also shown in Fig.18, which indicates that the data points except R 4286 showed an Arrehnius' relationship roughly and an averaged activation energy for three alloys was 290 KJ/mol.

4. DISCUSSIONS

The environment used in this report was heavy carburizing condition. Such environment might only be expected in chemical plants. However, for the comparative study of carburization resistance of alloys themselves, the test in this environment is quite efficient.

4.1 Carburization Kinetics

Concerning the carburization kinetics referred to the change in carbon content, it has been reported[12] that if no oxide is present on the alloy and surface reaction rates are sufficently high, the carburization rate will be controlled by diffusion and precipitation processes in the alloy matrix. Under such conditions, the carburization rate follows parabolic rate law. It has also been found[12] that assuming an activation energy of 209 KJ/mol for the carburization of Inconel 617, the parabolic rate constants for Inconel 617 and Incoloy 800 H at 950°C are approximately 0.2 and 2 $mg^2.cm^{-4}.h^{-1}$, respectively.

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In this investigation, it was found that the carburization rate of tested alloys which could not form oxide scale (except R 4286) followed parabolic rate law, and the parabolic rate constants for Hastelloy XR-II, Hastelloy XR and F alloy at 950°C were approximately 0.61, 0.67 and 0.47 $mg^2.cm^{-4}.h^{-1}$, respectively. From the facts mentioned above, it can be seen that the results reported here were quite reasonable. Furthermore, the parabolic rate constants of three alloys at 900°C are as follows ; Hastelloy XR-II = 0.19, Hastelloy XR = 0.22 and F alloy = 0.08 $mg^2.cm^{-4}.h^{-1}$.

When compared with the carburization resistance among three alloys, Hastelloy alloys XR and F alloy, F alloy exhibited better carburization behaviour. The results may be caused by the lower Cr content (higher carbon activity in the alloy) and the slower diffusion rate of carbon into the alloy matrix. Because it has been pointed out that the change in carbon content due to carburization for high temperature alloys increases with Cr content[13] and the diffusion rates of some alloying elements in Ni-Cr-W alloys matrixes are slower than that in Hastelloy alloys X matrixes[14].

4.2 Carburization Behaviour of R 4286

R 4286 was almost covered by oxide scale at each testing temperatures, so that emphasis is placed on the carburization behaviour of this alloy.

As can be seen from the carbon uptake and the temperature dependence curves, R 4286 showed different behaviour when compared with the other three alloys. As shown in Fig.s 5 and 6, the increase in carbon content at 900 and 950°C after 50 hours can be seen to saturate with time, which means that carbon transfer into the alloy matrix is completely stopped by the protective oxide layer. Such carburization behaviour has sometimes been observed on high temperature alloys during exposure in certain environment[15], in which surface of the alloy is covered by stable Cr-based oxide. Regarding the carburization resistance of R 4286, it can be concluded that Al-Ti-based oxide played a role of barrier against carbon transfer into the alloy matrix even in heavy carburizing environment, particularly the effect of barrier was effective at 950°C, i.e. Al-Ti-based oxide was more stable at 950°C.

It can be made clear from this investigation that if this alloy is used for the component material at high temperature parts in HTR, at least the carburization problem will be neglected in the environmental effects. This alloy, however, has two following demerits for HTR applications ;

(1) poor oxidation resistance in standardized simulated HTR environment like JAERI type B and PNP helium[9].

(2) higher Co content.

Cobalt may be an element which is best avoided for primary circuit applications in nuclear system because of the possibility of radioactive contamination. For HTR applications, therefore, the new alloy system based on R 4286 will be required. The new alloys are under discussion, in which for the improvement of oxidation resistance Mn and Si will be added, and Co will be replaced by Ni, Mo and W.

With respect to carburization behaviour, further investigations are in progress to establish the effect of carburization on mechanical properties of the carburized materials.

5. SUMMARY

Carburization tests of high temperature alloys, Hastelloy XR, Hastelloy XR -II, Ni-Cr-W (F) alloy and R 4286, were carried out in heavy carburizing environment at 800, 850, 900 and 950°C for up to 100 hours.

The results obtained showed that ;

(1) In the test conditions excpt at 800°C, in which Cr can not oxidize, Hastelloy alloys XR and F alloy were not covered by oxide scale, while for R 4286 Al-Ti-based oxide was formed on the specimens.

(2) The carburization kinetics of Hastelloy alloys XR and F alloy followed parabolic rate law, and the carbon uptake rate of F alloy was lower than those of Hastelloy alloys XR.

(3) R 4286 containing 2% Al and 2.5% Ti exhibited the best carburization resistance in the alloys tested, because additions of Al and Ti had a beneficial effect against carburization due to the formation of the protective Al-Ti-based oxide layer even in dry environment.

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Table 1 Chemical composition of the alloys tested (wt%).

Alloy	C	Si	Mn	P	S	Ni	Cr	W	Mo	Co	Fe	Al	Ti	Zr	B*
R4286	0.043	0.01	-	0.004	0.004	Bal.	18.14	5.50	4.15	10.60	0.05	1.99	2.49	0.059	35
Ni-Cr-W (F)	0.015	0.27	0.94	0.002	0.002	Bal.	18.14	21.62	-	0.01	-	0.009	0.25	0.05	2
Hastelloy XR-II	0.079	0.27	0.87	<0.005	0.001	Bal.	21.96	0.46	9.24	0.12	18.33	0.03	0.05	0.001	40
Hastelloy XR	0.077	0.34	0.84	<0.005	<0.005	Bal.	21.67	0.48	8.97	0.05	18.24	<0.05	<0.05	-	<10

* ppm , - not determined

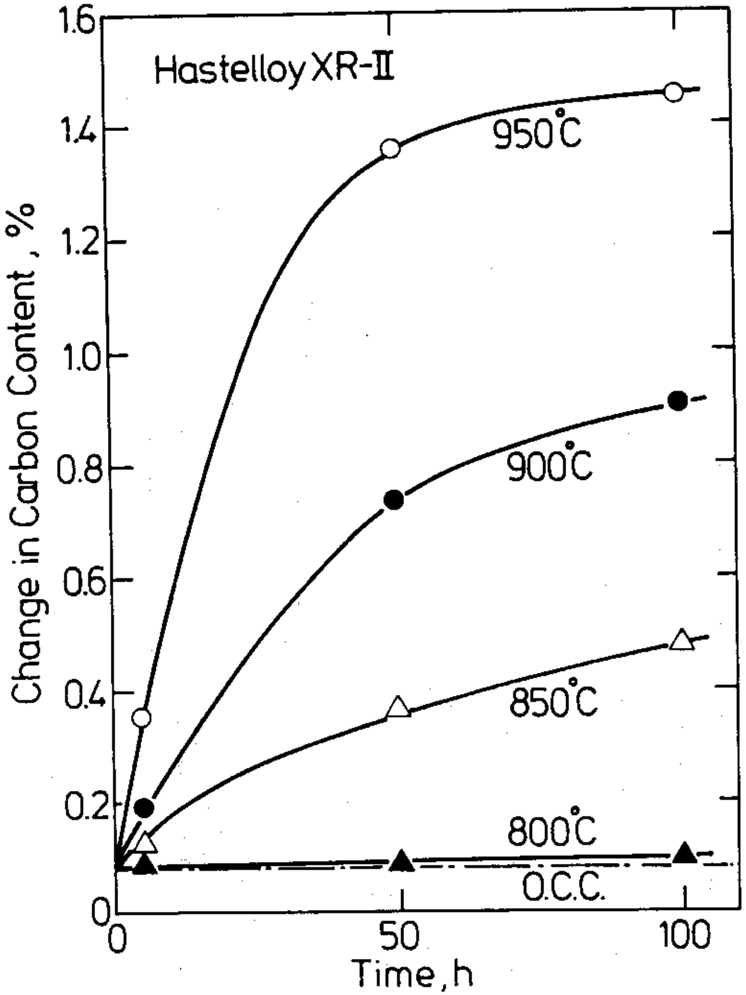

Fig. 1 Change in carbon content of Hastelloy XR-II at 800, 850, 900 and 950°C.
(O.C.C. : Original Carbon Content)

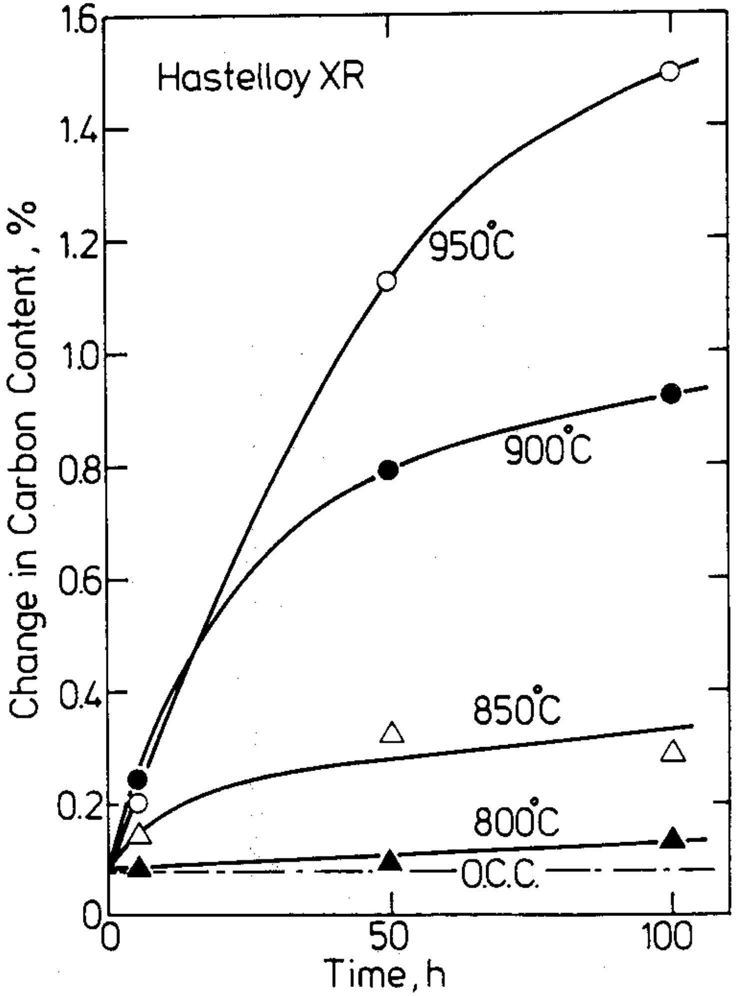

Fig. 2 Change in carbon content of Hastelloy XR.

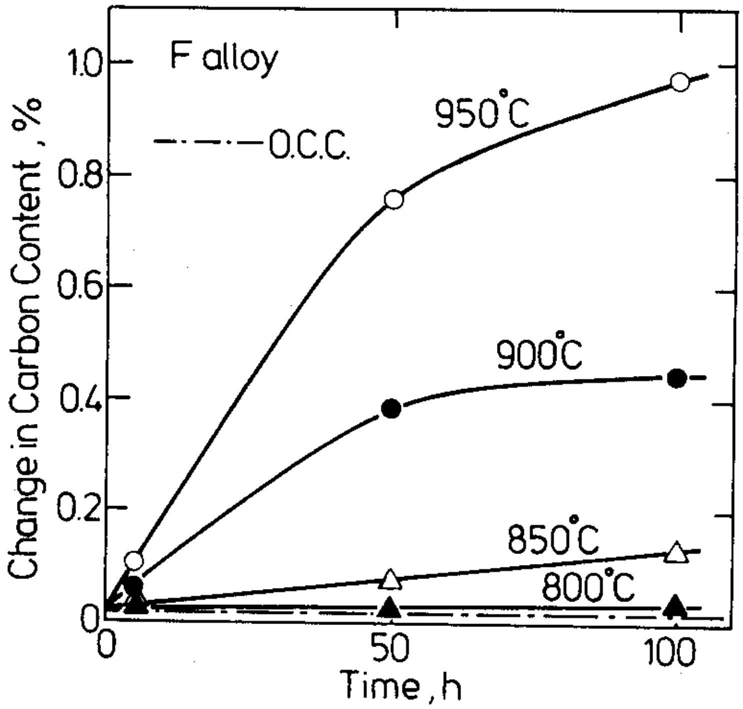

Fig. 3 Change in carbon content of F alloy.

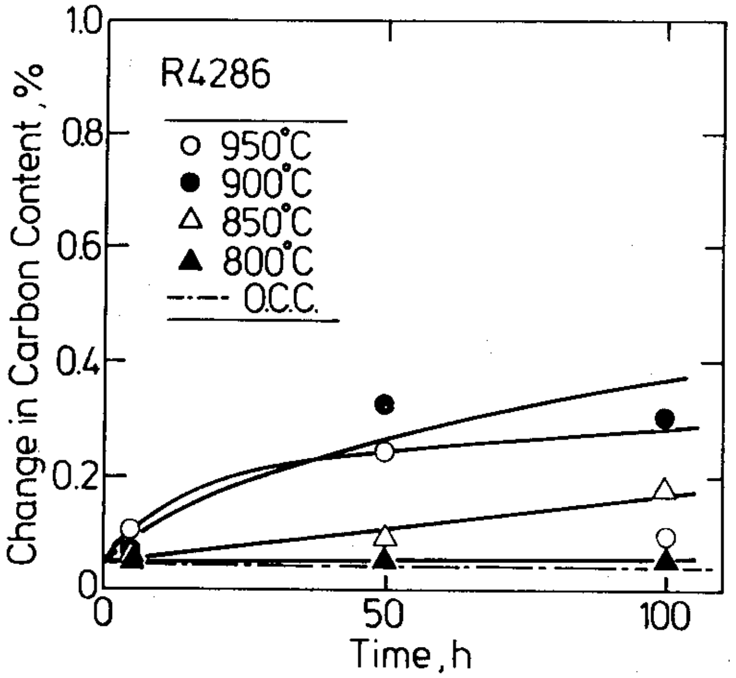

Fig. 4 Change in carbon content of R4286.

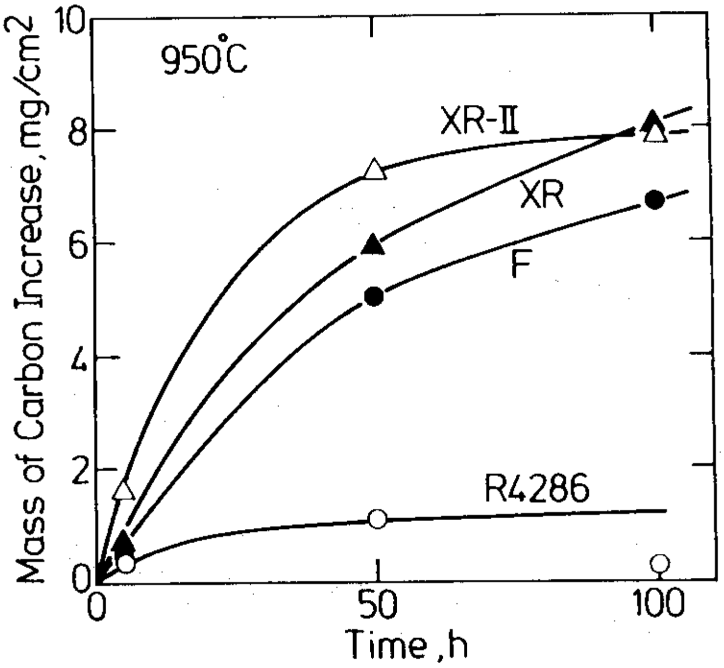

Fig. 5 Carburization kinetics of the alloys tested at 950°C.

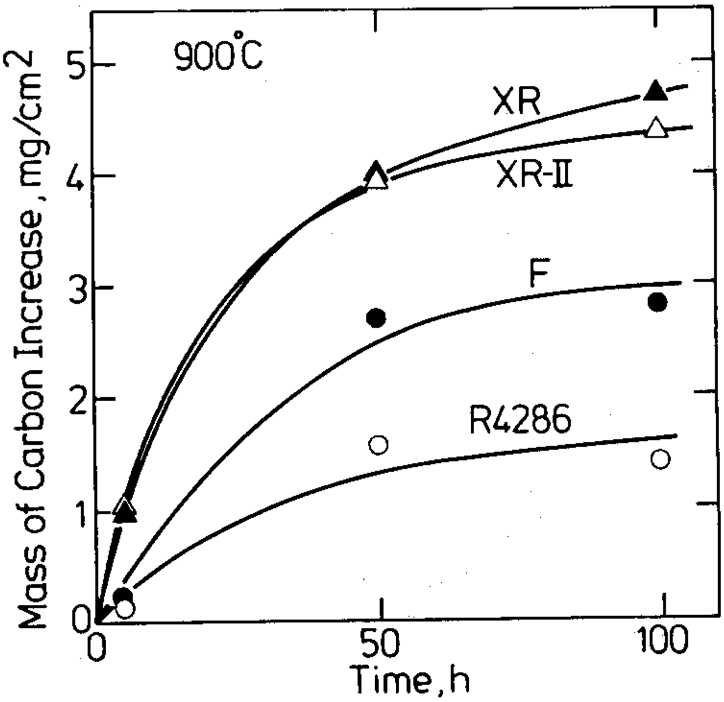

Fig. 6 Carburization kinetics of the alloys tested at 900°C.

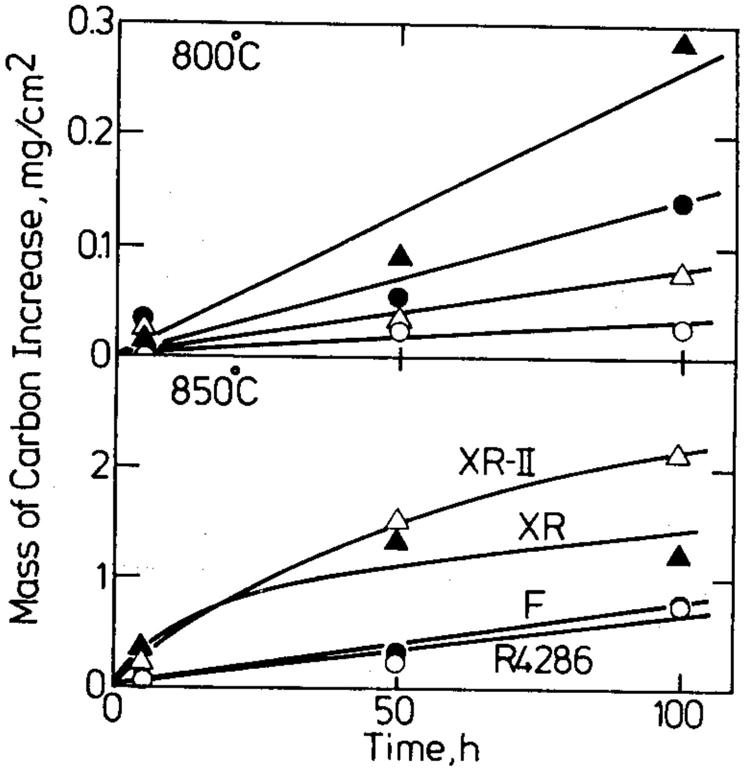

Fig. 7 Carburization kinetics of the alloys tested at 850 and 800°C.

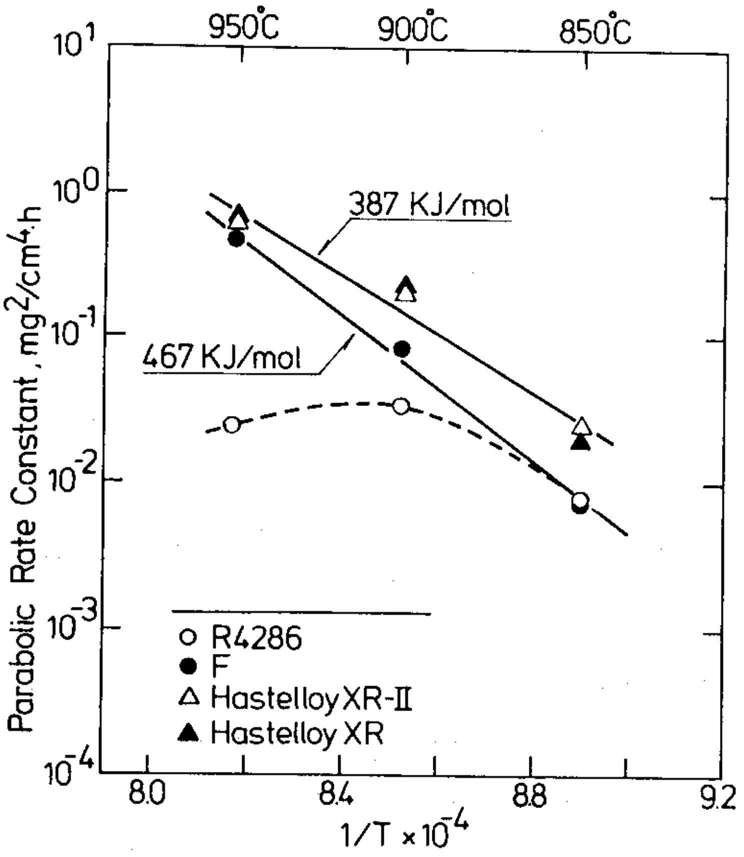

Fig. 8 Temperature dependence of parabolic rate constants for change in carbon content.

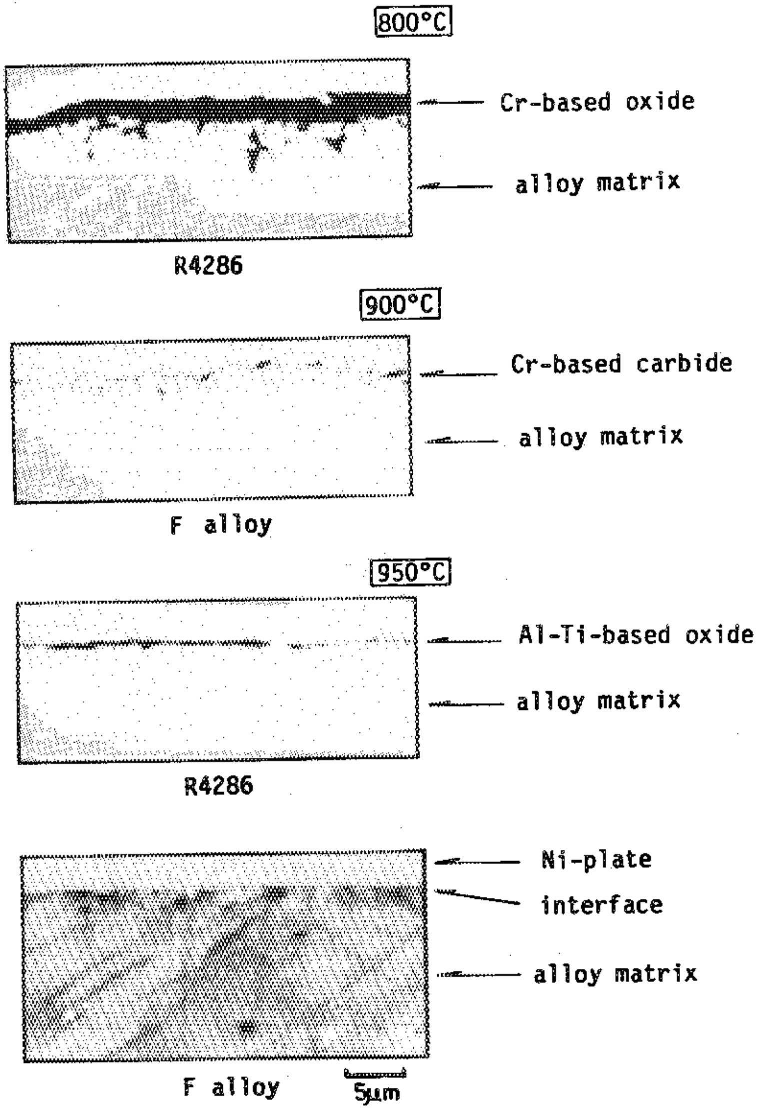

Fig. 9 Results of qualitative analysis of surface products at several temperature.

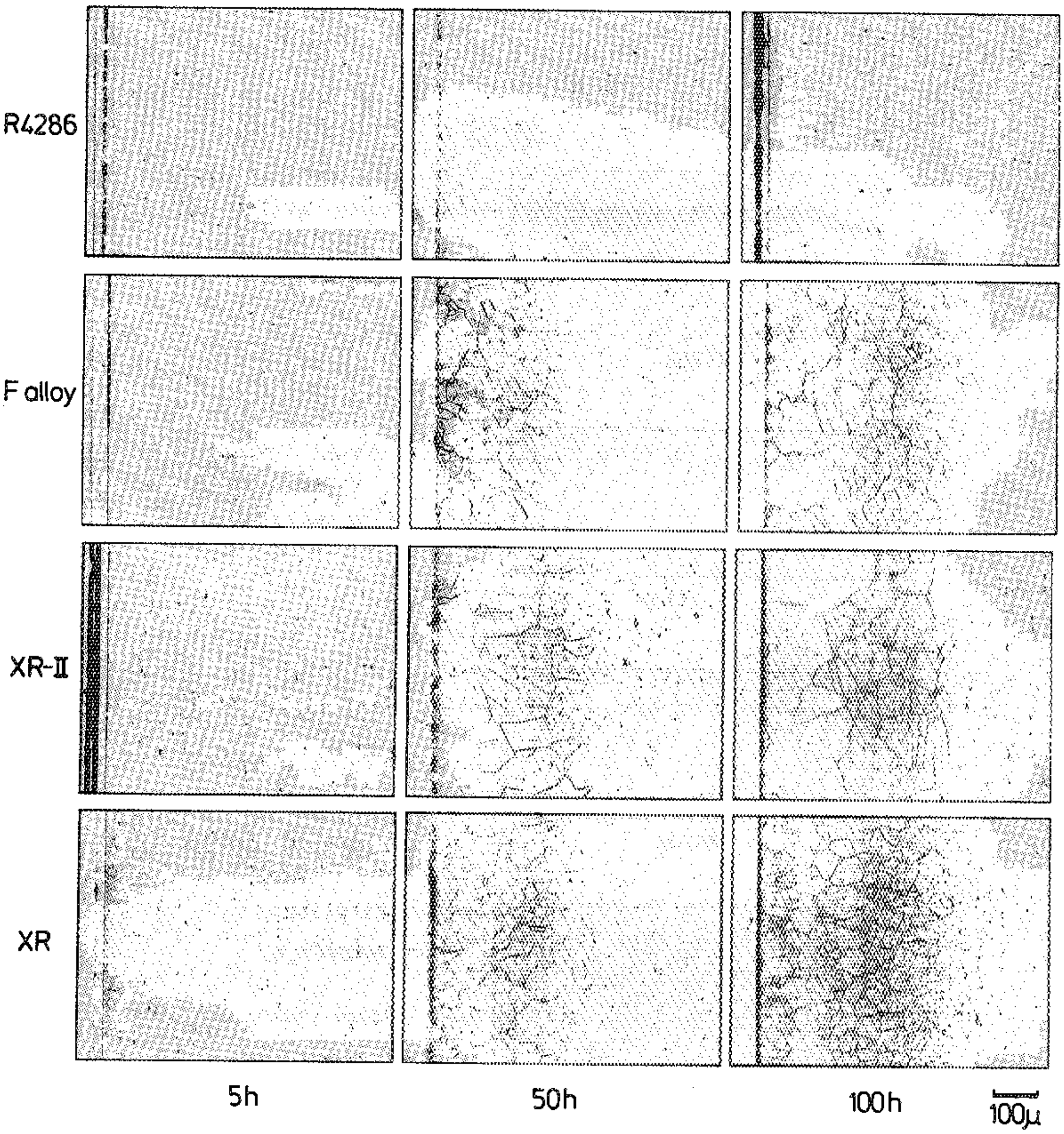

Fig. 10 Carburized microstructures of the alloys tested at 950°C for 100 h (no etching).

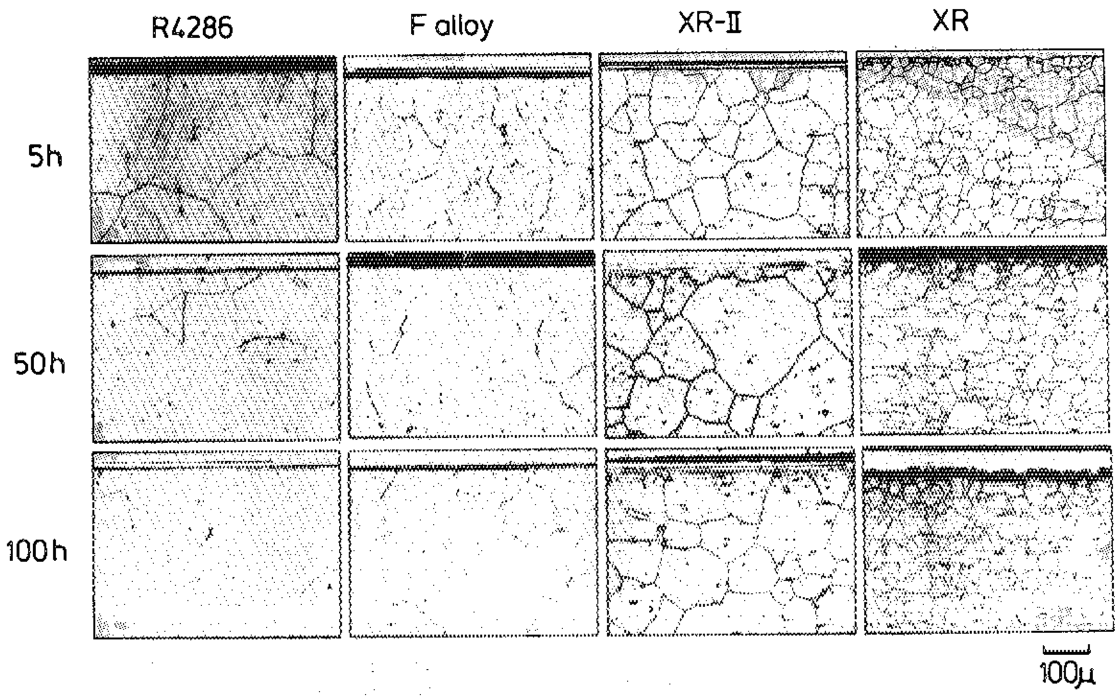

Fig. 11 Microstructures of the alloys tested at 800°C (etching).

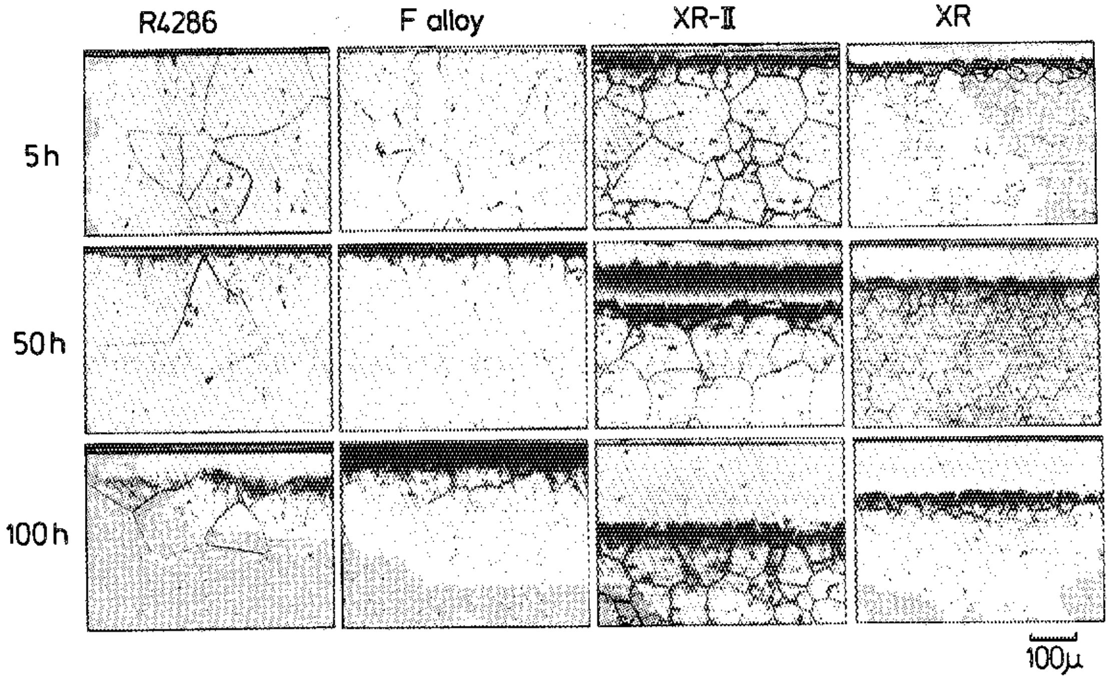

Fig. 12 Micristructures of the alloys tested at 850°C (etching).

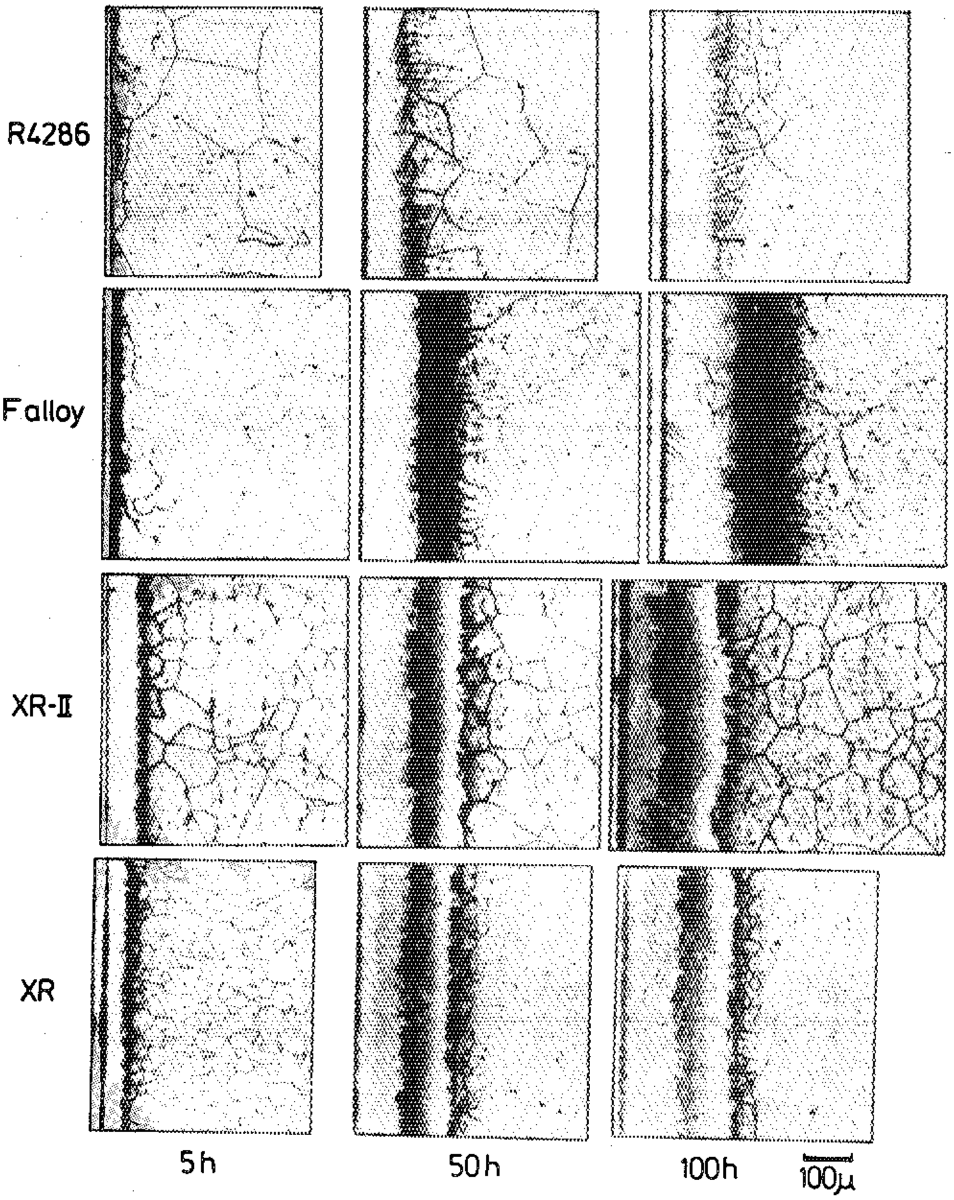

Fig. 13 Microstructures of the alloys tested at 900°C (etching).

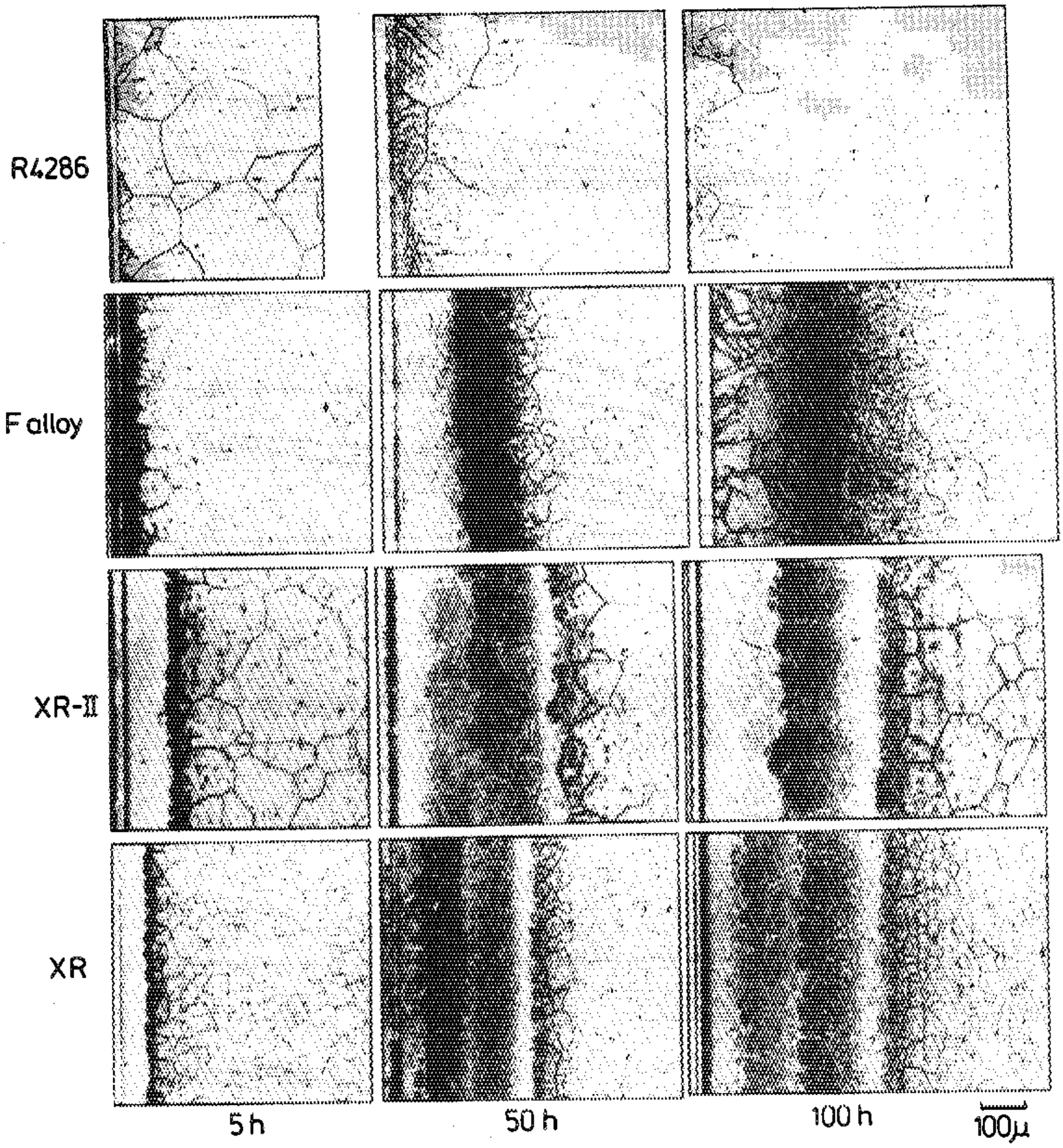

Fig. 14 Microstructures of the alloys tested at 950°C (etching).

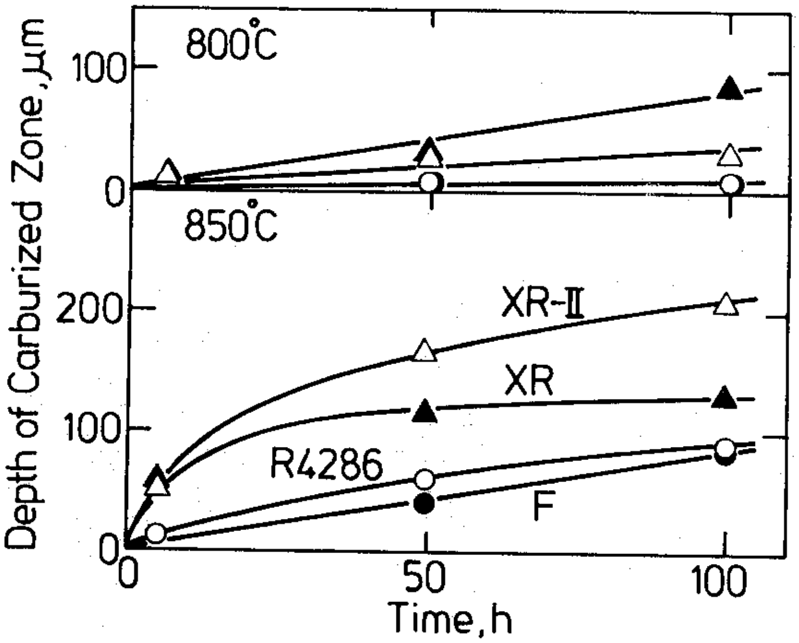

Fig. 15 Depth of carburized zone of the alloys tested at 800 and 850°C.

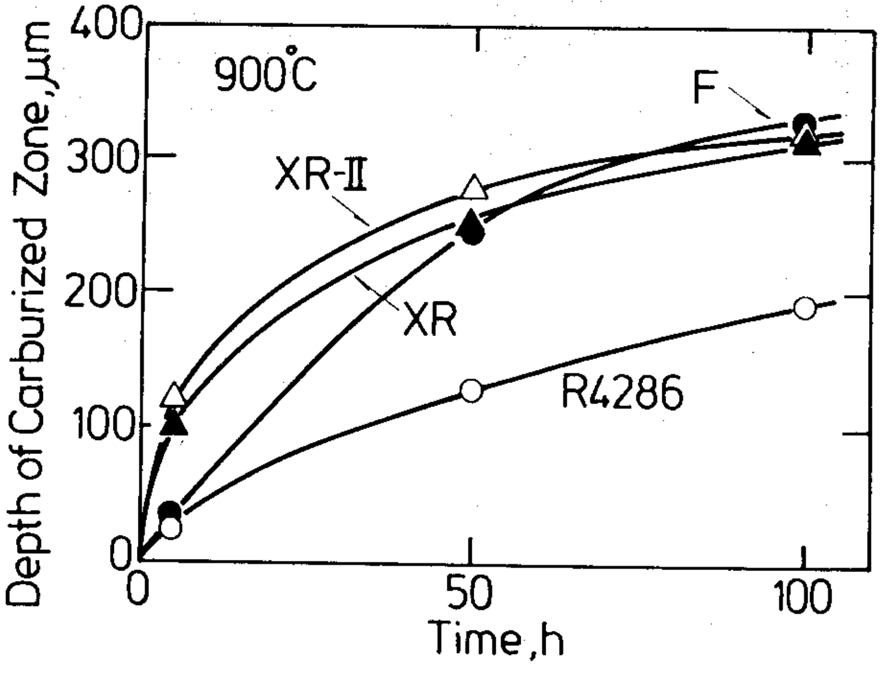

Fig. 16 Depth of carburized zone of the alloys tested at 900°C.

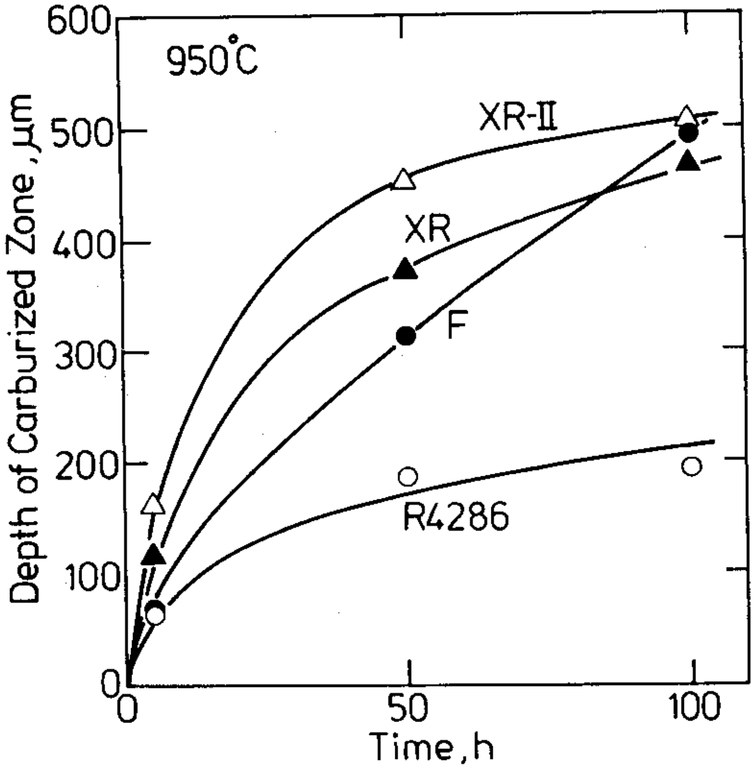

Fig. 17 Depth of carburized zone of the alloys tested at 950°C.

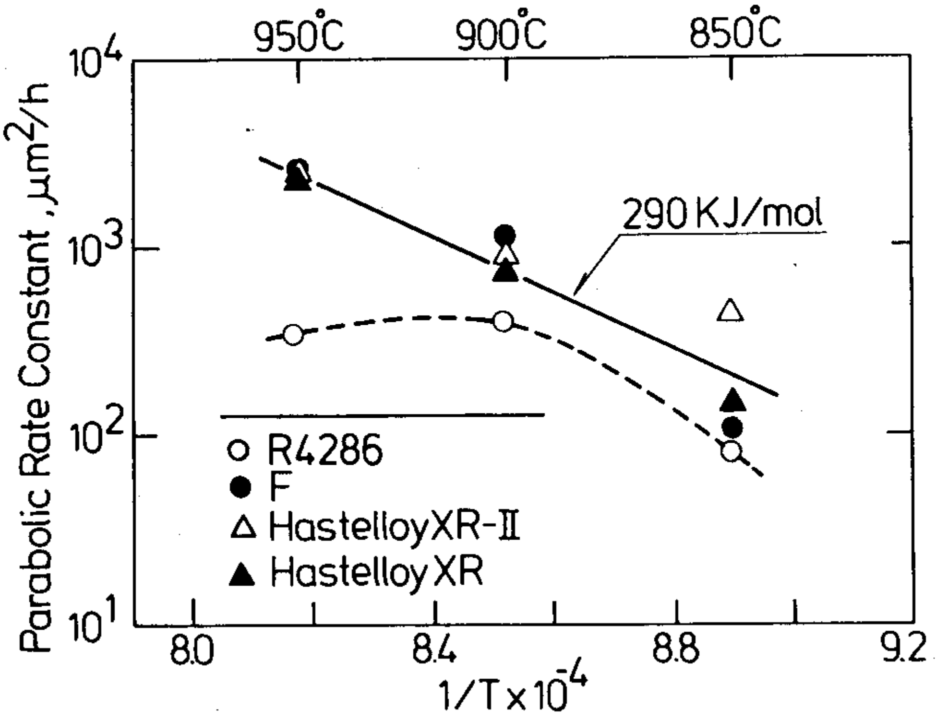

Fig. 18 Temperature dependence of parabolic rate constants for depth of carburized zone.