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Abstract: Phase transformation and high-temperature shape memory effect of Ti(Pt, Ir) were investigated. First, the Ti-rich phase boundary of Ti(Pt, Ir) was investigated by phase composition analysis by secondary electron microscopy (SEM) using an electron probe X-ray micro analyzer (EPMA), X-ray diffraction analysis and transmission electron microscopy (TEM). Then, the three alloys Ti-35Pt-10Ir, Ti-22Pt-22Ir, and Ti-10Pt-32Ir (at%) close to the phase boundary but in the single phase of Ti(Pt, Ir) were prepared by the arc melting method. The shape memory effect and lattice parameter of Ti(Pt, Ir) was investigated by compression loading-unloading tests and high-temperature X-ray diffraction analysis, respectively. The composition dependence of martensitic phase transformation temperature, crystal structure, and shape memory effect was investigated in Ti-Pt-Ir.

Composition dependence of phase transformation behavior and shape memory effect of  
TiPt

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(1) Fig.3, the tie lines are deviated from the correct positions between gray and white circles.

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(2) P.5, line 6, "the small entropy for..." -> "the small entropy change for..."

I changed to "the small entropy change for "

(3) P.5, line 8, Please insert the numerical data of  $L_0$ ,  $L'$  and  $L''$ .

It is difficult to indicate the numerical data of  $L_0$ ,  $L'$  and  $L''$ , then I inserted table 2.

(4) P.6, line 14, "840, 910 for ..." -> "840, 910 MPa for ..."

I changed to "the small entropy change for "

Composition dependence of phase transformation behavior and shape memory effect of Ti(Pt, Ir)

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Abstract

The phase transformation and high-temperature shape memory effect of Ti(Pt, Ir) were investigated. First, the Ti-rich phase boundary of Ti(Pt, Ir) was investigated by phase composition analysis by secondary electron microscopy (SEM) using an electron probe X-ray micro analyzer (EPMA), X-ray diffraction analysis and transmission electron microscopy (TEM). Then, the three alloys Ti-35Pt-10Ir, Ti-22Pt-22Ir, and Ti-10Pt-32Ir (at%) close to the phase boundary but in the single phase of Ti(Pt, Ir) were prepared by the arc melting method. The shape memory effect and crystal structure were investigated by compression loading–unloading tests and high-temperature X-ray diffraction analysis, respectively.

Keywords

high-temperature alloys, intermetallics, shape memory, SEM, X-ray diffraction

Highlight

The high-temperature shape memory effect of Ti(Pt, Ir) was investigated. The shape recovery ratio was 72% in Ti-10Pt-32Ir after deformation at 1123 K.

## I. Introduction

TiNi-based shape memory alloys are already in practical use, and the next stage is to develop high-temperature shape memory alloys (HTSMAs) for gas turbines, rocket engines, automotive engines, nuclear reactors, and so forth. This will require a higher martensitic transformation temperature. Several alloy systems have been proposed [1], including the addition of refractory metals such as Hf and Zr, and platinum group metals such as Pt and Pd, to TiNi. A significant body of data exists for Ti-Ni-Pd alloys, indicating acceptable work performance up to 35 at% Pd. The martensitic transformation temperature at 35 at% Pd is about 573 K, and it increases with higher Pd content [2]. However, at compositions above 35 at% Pd, recovery and recrystallization occur at operating temperatures in the range of 450 – 600°C and irrecoverable strain becomes large [3], making it difficult to develop HTSMAs with Pd contents above about 35 at%. Therefore, research is now focusing on reducing irrecoverable strain in Ti-Ni-Pd alloys with Pd content of less than 35 at%. One advantage of Ti-Ni-Pt alloys with Pt content over 30 at% is that the martensitic transformation temperature is high above 773 K [4-6], but then deformation by creep becomes severe [7]. Similar to Ti-Ni-Pd, the desirable composition of Ti-Ni-Pt is considered to be below 30 at%.

Although Ti-Ni-Pd/Pt alloys still need to be improved, the addition of Pd and Pt is promising. We have focused on TiPt-based shape memory alloys [8-14]. In Ti-Pt alloys, the B2 parent phase (cubic structure) transforms to the B19 martensite phase (orthorhombic structure) and the transformation temperature of TiPt at equiatomic composition is about 1323 K [15]. Ir was chosen as a solid solution hardening element for TiPt, and the phase transformation and strain recovery were investigated for  $\text{Ti}_{50}(\text{Pt}, \text{Ir})_{50}$  [8, 9, 11, 13]. Transformation temperature increased by Ir addition and was found to be 1491 K in Ti-12.5Pt-37.5Ir [8, 11]. The shape memory effect of  $\text{Ti}_{50}(\text{Pt}, \text{Ir})_{50}$  was investigated by the thermal expansion test and compression test [11]. Strain recovery of 1.7% and recovery ratio of 57% were obtained in Ti-25Pt-25Ir after deformation at 1123 K followed by heat treatment above the transformation temperature, although recovery was not perfect. The change in crystal structure by addition of Ir was investigated by high-temperature X-ray diffractometry, and it was found that the structure changed from orthorhombic to almost tetragonal in the above composition range. An increase of tetragonality is thought to facilitate phase transformation, resulting in relatively good shape recovery [14]. However, although several alloy compositions have been tried, perfect recovery has not been achieved. Another question is how the B2 phase region extends in the Ti-Pt-Ir ternary system and how the addition of Ir changes the transformation temperature. In the Ir-Ti binary phase diagram, the transformation temperature is high for Ir-rich composition, but drastically decreases with increase of Ti content. When a large amount of Ir is added to Ti-Pt, the transformation temperature in the Ti-rich region may be decreased drastically.

In this study, first the phase boundary of the B2 phase was investigated in the Ti-rich Ti-Pt-Ir ternary system. Based on the obtained phase boundary, several alloys with Ti-rich composition were prepared. The composition dependence of martensitic phase transformation temperature, crystal structure, and shape recovery was investigated.

## II. Experimental procedure

Six kinds of 10-g alloy ingots with nominal composition of Ti-35Pt-10Ir, Ti-22Pt-22Ir, Ti-10Pt-32Ir,

Ti-27Pt-13Ir, Ti-20Pt-20Ir and Ti-13Pt-27Ir (at%) were produced by the arc melting method. Ti-27Pt-13Ir, Ti-20Pt-20Ir and Ti-13Pt-27Ir are alloys with Ti content of 60 at%. These ingots were sealed in a silica tube with a small amount of Ar gas and heat treated for 168 hours and/or 3 hours depending on the alloy composition at 1523 K, a higher temperature than  $A_f$ . After heat treatment, the ingots were ice-water quenched.

Plate samples were sliced from the heat-treated ingot for microstructure observation. The sliced samples were embedded in resins and mechanically polished using sandpaper and diamond. The back-scattered images were taken at an accelerating voltage of 15 kV in a FE-SEM, JEOL 7001F. Phase composition was investigated in FE-SEM equipped with EPMA (JEOL JXA8900R). An accelerating voltage of 15 kV with a stabilized beam current of  $1 \times 10^{-7}$  A was used. Ti was analyzed using Ti-K $\alpha$  S-ray lines with an LIF as the analyzing crystal, and Ir and Pt were analyzed using Ir-M $\alpha$  and Pt-M $\alpha$  X-ray lines with PETH as the analyzing crystal.

Sliced samples of 3 mm in diameter were cut from the heat-treated ingot for microstructure observation using TEM. The sliced samples were mechanically polished using sandpaper and ion milled, and the microstructure was observed at an accelerating voltage of 200 kV by TEM (Tecnai 20).

Small pieces measuring  $2 \times 2 \times 0.8$  mm were cut from button ingots and the phase transformation temperature was investigated by differential thermal analysis (DTA) between 473 and 1673 K at a heating and cooling rate of 0.17 K/s using a Shimadzu DTA-50.

Powder samples of micron order were prepared for Ti-35Pt-10Ir, Ti-22Pt-22Ir, and Ti-10Pt-32Ir by mechanically crushing heat-treated ingots for high-temperature X-ray diffraction analysis. The phase constituent was investigated at temperatures from room temperature to 1573 K using a Rigaku TTR-III high-temperature X-ray diffractometer equipped with Cu K $\alpha$  radiation. The powder samples were placed on an Al<sub>2</sub>O<sub>3</sub> plate in the Pt sample holder and heated in a vacuum furnace with a Pt heater. A Pt-Rh thermocouple was placed under the Pt holder to measure and control the temperature. The heating rate was 40 K/min and the samples were kept at the testing temperature for 5 min before X-ray analysis. The measurements from room temperature to 1573 K were conducted twice to remove plastic deformation obtained during preparation of powder samples by crushing.

Plate samples measuring  $8 \times 8 \times 1$  mm were used to investigate the structure of Ti-27Pt-13Ir, Ti-20Pt-20Ir, and Ti-13Pt-27Ir. The phase constituent was investigated at room temperature using a Rigaku 2500 equipped with Cu K $\alpha$  radiation.

Rectangular samples measuring  $2.5 \times 2.5 \times 5$  mm were cut from the ingots using an electrical discharge machine for the compression test. The loading–unloading compression test was carried out at an initial strain rate of  $3 \times 10^{-4}$ /s at 1123 K using the Shimadzu AG-I test system. After the compression test, samples were heated at 1523 K for 1 h and the shape memory strain was measured by comparing the sample length before and after heat treatment.

### III. Results and Discussion

#### 1. Phase boundary of the martensite phase

Microstructures of Ti-27Pt-13Ir, Ti-20Pt-20Ir, and Ti-13Pt-27Ir alloys observed after heat treatment at



1523 K for 168 hours followed by ice-water quenching are shown in Fig. 1. Two phases, a dark phase and a bright phase, were observed in all three alloys as shown in Figs. 1a–1c. In the enlarged microstructure in Figs. 1d–1f, several straight lines similar to a twin structure were observed in the bright phase although the contrast is not clear. These straight lines suggest that the bright phase is the martensite phase. Phase compositions investigated by EPMA are summarized in Table 1. As expected from the microstructure in Figs. 1d–1f, the composition of the bright phase is close to the martensite phase composition of Ti:60 and Pt+Ir:40. The composition of the dark phase was close to Ti:70 and Pt+Ir:30. According to the phase diagram of the Ti-Pt and Ti-Ir binary system [15], intermetallic compounds such as  $Ti_3Pt$  and  $Ti_3Ir$  appear in the composition range 72–77 at% Ti for Ti-Pt and 73–75 at% Ti for Ti-Ir. X-ray diffraction analysis was conducted to confirm the phase structure for the heat-treated plate samples. In all the tested alloys, diffraction patterns from the martensite B19 phase were observed as shown in Fig. 2 and the bright phase can thus be identified as B19 phase. This indicates that the B2-parent phase at 1523 K was transformed to the B19-martensite phase during quenching. Peaks from the dark phase did not appear for some reason as shown in Fig. 2 despite the existence of several unknown peaks. In the  $A_{50}B_{50}$  compound, the tetragonal  $L1_0$  is also a possible structure. For example, the  $L1_0$  structure was found in IrTi at 1273 K where Ir is one of the platinum group metals the same as Pt [16]. Therefore, to identify unknown peaks, the  $L1_0$  structure was also considered. In Fig. 2(c), square symbols represent the peak position of the  $L1_0$  structure; it can be seen that some unknown peaks fitted the peak position of the  $L1_0$  structure. However, peaks at a high angle of the  $L1_0$  structure did not clearly appear in our experimental results. In previous studies, we investigated the microstructure of Ti-50(Pt, Ir) by TEM, but found no clear evidence for the existence of the  $L1_0$  structure [17, 18]. Thus, the  $L1_0$  structure may exist, but has not been confirmed yet. To investigate the structure of the dark phase, the diffraction pattern of the dark phase was investigated by TEM. The crystal structure of  $Ti_3Pt$  and  $Ti_3Ir$  is the same as the  $Cr_3Si$  type cubic structure of the space group of #223 and the lattice parameters are 5.03 Å for  $Ti_3Pt$  and 5.00 Å for  $Ti_3Ir$  [19]. The lattice parameters of  $Ti_3Pt$  and  $Ti_3Ir$  are close to each other and thus it is considered that  $Ti_3Pt$  and  $Ti_3Ir$  will fully mix with each other. The observed diffraction patterns indicate that the dark phase is the same as the  $Cr_3Si$  type structure with lattice parameter of 4.952 Å, suggesting that the dark phase is  $Ti_3(Pt, Ir)$ .

These results show that the  $Ti_3(Pt, Ir)$  phase equilibrates with the Ti(Pt, Ir) martensite phase. The schematic partial isothermal section using the phase composition at 1523 K is drawn in Fig. 3. To investigate the shape memory behavior of Ti-rich Ti-Pt-Ir alloys, alloys with compositions of Ti-35Pt-10Ir, Ti-22Pt-22Ir, and Ti-10Pt-32Ir indicated by triangle symbols were newly prepared.

## 2. Shape memory effect of Ti-rich Ti-Pt-Ir alloys

Microstructures of Ti-35Pt-10Ir, Ti-22Pt-22Ir, and Ti-10Pt-32Ir heat treated at 1523 K for 3 hours, followed by ice-water quenching are shown in Fig. 4. As expected from the isothermal section at 1523 K, a typical martensite structure appeared in the whole area of the samples. The multi-variant martensite with lens type shape formed in Ti-35Pt-10Ir as shown in Fig. 4a, but a single variant martensite with straight line shape formed in the other two alloys as shown in Fig. 4b and 4c. The dark phases observed along grain boundaries in Fig. 4a and 4b are considered to be titanium oxide because oxygen peaks appeared during

phase composition analysis, although a detailed analysis is necessary.

Phase transformation temperature was investigated using DTA. However, no sharp peak was observed except for Ti-35Pt-10Ir. Only Ti-35Pt-10Ir indicated a small peak during heating, even though no peaks were observed during cooling. The austenite starting temperature,  $A_s$ , was 1204 K and finishing temperature,  $A_f$ , was 1232 K obtained from the peaks during heating, which are 100 K lower than those of Ti-50at%Pt. The reason for the small or undetectable peaks of DTA is the small entropy change for phase transformation. To investigate phase transformation temperature, high-temperature X-ray diffraction analysis was performed from room temperature to 1573 K. The X-ray diffraction pattern of Ti-35Pt-10Ir is shown in Fig. 5. Peaks from the B19 phase clearly appeared up to 1273 K. At 1473 and 1573 K, strong peaks from the parent B2 phase were observed, but peaks from the B19 phase were also obtained together with the peaks of the B2 phase. The X-ray diffraction pattern indicates that the transformation temperature lies between 1273 and 1473 K. The difference between phase transformation temperature obtained by DTA and high-temperature XRD was about 100 K. It is considered that the sample temperature of XRD is 100 K lower than the controlling temperature due to radiation from the sample surface. XRD diffraction patterns of the other two alloys were also the same as that of Ti-35Pt-10Ir. Since these two alloys did not show any peak in DTA, the phase transformation temperature for Ti-rich Ti-Pt-Ir alloys is not clear yet. However, from high-temperature XRD, the martensite structure was stable at least up to 1173 K. When the sample was quenched, it seems that phase transformation occurred perfectly because the martensite structure formed in the whole of the samples. On the other hand, reverse transformation did not occur perfectly during heating since the peaks from the martensite phase appeared after the peaks from the parent phase appeared in high-temperature XRD results. In our previous study, reverse transformation occurred perfectly in Ti-50(Pt, Ir), but the reason for the incomplete reverse transformation in Ti-rich Ti(Pt, Ir) in this study is not clear.

Similar to Fig. 2, there are some unknown peaks in Fig. 5 but there are more of them. One possibility is the coexistence of the  $L1_0$  structure because some peaks fitted the  $L1_0$  structure, while another possibility is that the stacking order of the B19 structure is locally changed [17, 20, 21]. Further investigation is necessary.

Since the phase transformation temperatures of Ti-rich alloys are considered to be above 1173 K, compression tests were performed at 1123 K in the martensite state. The residual strain  $\varepsilon_r$  after unloading was estimated from the initial sample length  $L_0$  and the deformed sample length  $L'$  measured at room temperature using the following equation:

$$\varepsilon_r = (L_0 - L')/L_0 \quad (1)$$

To investigate shape recovery, the deformed sample was heated above  $A_f$  at 1573 K in the parent phase for 1 hour, followed by cooling. The recovered sample length  $L''$  was measured after cooling at room temperature and the irrecoverable strain  $\varepsilon_{irr}$  was assessed by comparing the recovered sample length  $L''$  and the initial sample length  $L_0$  using the equation:

$$\varepsilon_{irr} = (L_0 - L'')/L_0 \quad (2)$$

Then, the shape memory strain  $\varepsilon_{sme}$  was obtained by subtracting the irrecoverable strain  $\varepsilon_{irr}$  from the residual strain  $\varepsilon_r$  after unloading using the equation:

$$\varepsilon_{sme} = \varepsilon_r - \varepsilon_{irr} \quad (3)$$

Thus, the strain recovery ratio is defined by:

$$\text{Strain recovery ratio} = \varepsilon_{sme} / (\varepsilon_{sme} + \varepsilon_{irr}) \quad (4)$$

The initial sample length  $L_0$ , the deformed sample length  $L'$ , and the recovered sample length  $L''$  are summarised in Table 2. The strain recovery ratio is plotted as a function of Ir content together with the previous results as shown in Fig. 6. Strain recovery increased with higher Ir content. The highest strain recovery ratio of 72% was obtained in Ti-10Pt-32Ir and the recovery strain was 1.4% despite incomplete reverse transformation. In the previous study on alloys with Ti content of 50 at%, a relatively large strain recovery ratio of 50–60% was obtained in the alloys with Ir content of 10–30% as shown in Fig. 6 [14]. The highest strain recovery ratio, 57%, was obtained in Ti-25Pt-25Ir. The strain recovery of the most Ti-rich alloys and the alloys with Ti content of 50 at% was greater than that of TiPt binary compound whose strain recovery was 11%. The yield strength of the parent and martensite phases increased with increase of Ir content in Ti-50(Pt, Ir) [11]. Therefore, Ir-strengthening in Ti-rich Ti(Pt, Ir) is also expected to improve shape memory behavior. The effect of Ir addition for 0.2% flow stress was investigated from the stress–strain curves of the compression test in the martensite state. It was about 300 MPa for TiPt and 960, 840, 910 MPa for Ti-35Pt-10Ir, Ti-22Pt-22Ir, and Ti-10Pt-32Ir, respectively. This indicates that the addition of Ir in Ti-rich alloys also improves the high-temperature strength, resulting in improving shape recovery.

RuNb and RuTa have also been noted as high-temperature shape memory alloys [22, 23]. Like Pt and Ir, Ru is a platinum group metal. A two-step phase transformation, from the  $\beta$  phase with B2 structure to the  $\beta'$  phase with tetragonal structure and from the  $\beta'$  phase to the  $\beta''$  phase with monoclinic structure, appears in RuNb and RuTa. The shape memory effect was observed for the phase transformation from the  $\beta$  to  $\beta'$  phases, whose phase transformation temperature is 1393 K for  $\text{Ru}_{50}\text{Ta}_{50}$  and 1158 K for  $\text{Ru}_{50}\text{Nb}_{50}$ . The phase transformation temperature of RuTa is similar to that of Ti(Pt, Ir). Strain recovery of RuTa measured by a compression test at 1173 K was 2% [22], higher than 1.4% in Ti-10Pt-32Ir in this study. Since the strain recovery of binary TiPt after the compression test at 1123 K was only 0.2% [13], a suitable Ir content should be found in order to improve the strain recovery of TiPt to be comparable with that of RuTa.

#### IV. Conclusions

The Ti-rich phase boundary of Ti(Pt, Ir) was investigated by phase composition analysis by EPMA, X-ray diffraction analysis and TEM observation. The partial isothermal section at 1523 K was determined. Based on the partial isothermal section, the three alloys Ti-35Pt-10Ir, Ti-22Pt-22Ir, and Ti-10Pt-32Ir close to the phase boundary but in the single phase Ti(Pt, Ir) were prepared by the arc melting method. Martensite phase was formed in the whole area of the alloys, but reverse transformation was not perfect, as shown by high-temperature X-ray diffraction analysis. The shape recovery ratio was investigated for deformation at 1123 K followed by heat treatment in the parent phase. The shape recovery ratio increased

with Ir and the highest value, 72%, was obtained in Ti-10Pt-32Ir.

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## Captions

Table 1 Phase composition of Ti-Pt-Ir alloys analyzed by EPMA.

Table 2 The initial sample length  $L_0$ , the deformed sample length  $L'$ , and the recovered sample length  $L''$  of Ti-Pt-Ir.

Fig. 1 Backscattered images of (a) Ti-27Pt-13Ir, (b) Ti-22Pt-22Ir, and (c) Ti-13Pt-27Ir alloys heat treated at 1523 K for 100 hours followed by ice-water quenching. Enlarged backscattered images of (d) Ti-27Pt-13Ir, (e) Ti-22Pt-22Ir, and (f) Ti-13Pt-27Ir alloys heat treated in the same condition of (a-c).

Fig. 2 X-ray diffraction patterns taken at room temperature of (a) Ti-13Pt-27Ir, (b) Ti-22Pt-22Ir, and (c) Ti-27Pt-13Ir, respectively.

Fig. 3 Partial isothermal section at 1523 K. Black, gray, and white circles represent alloy composition and measured composition from two phases, respectively. Triangle symbols represent prepared alloy composition to investigate the shape memory effect.

Fig. 4 Backscattered images of (a) Ti-35Pt-10Ir, (b) Ti-20Pt-20Ir, and (c) Ti-10Pt-32Ir alloys heat treated at 1523 K for 3 hours followed by ice-water quenching.

Fig. 5 X-ray diffraction patterns of Ti-35Pt-10Ir from room temperature to 1573 K.

Fig. 6 Shape recovery ratio as a function of Ir content. Solid and open symbols represent Ti-rich alloys and the alloy with 50 at% Ti [14].

**Highlight**

The high-temperature shape memory effect of Ti(Pt, Ir) was investigated. The shape recovery ratio was 72% in Ti-10Pt-32Ir after deformation at 1123 K.

Table 1

	Bright phase (at%)			Dark phase (at%)		
	Ti	Pt	Ir	Ti	Pt	Ir
Ti-27Pt-13Ir	57.1	29.1	13.8	69.0	22.6	8.4
Ti-20Pt-20Ir	58.2	21.6	20.2	68.7	19.3	12.0
Ti-13Pt-27Ir	59.8	13.9	26.3	69.7	15.3	15.0

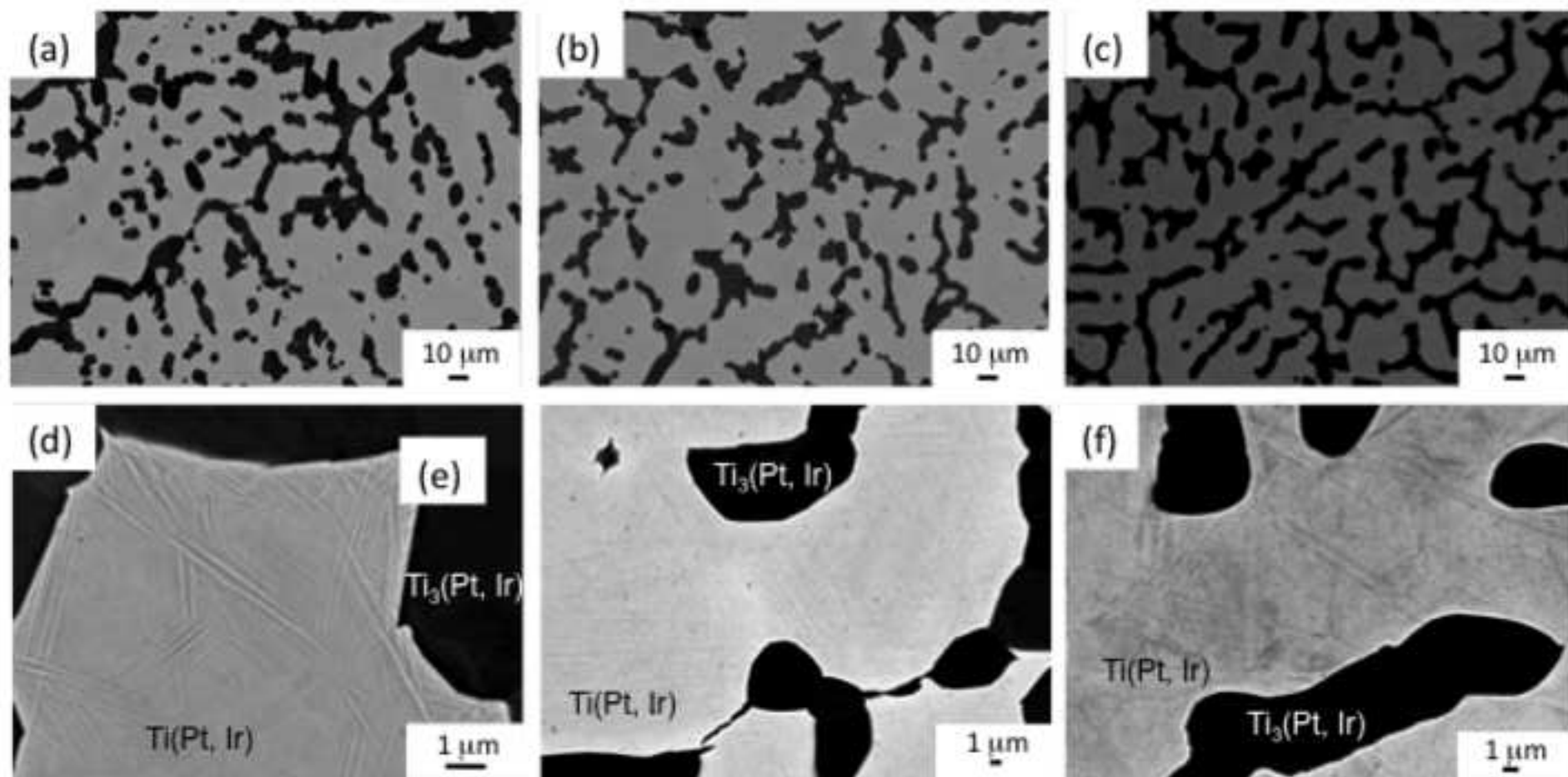
Table 2

	$L_0$	$L'$	$L''$
Ti-35Pt-10Ir	5.026	4.781	4.832
Ti-22Pt-22Ir	5.036	4.910	4.985
Ti-10Pt-32Ir	5.031	4.936	5.005

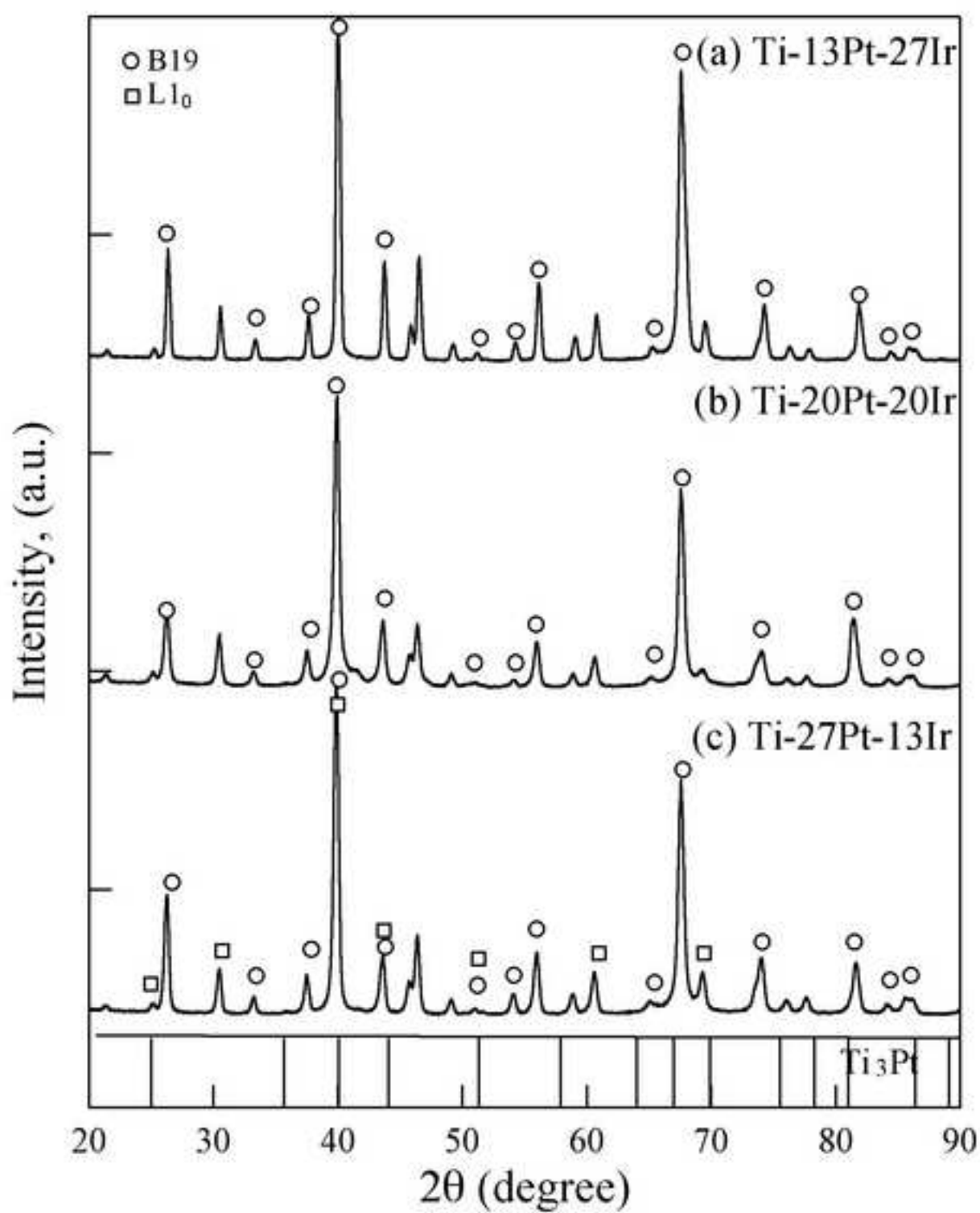


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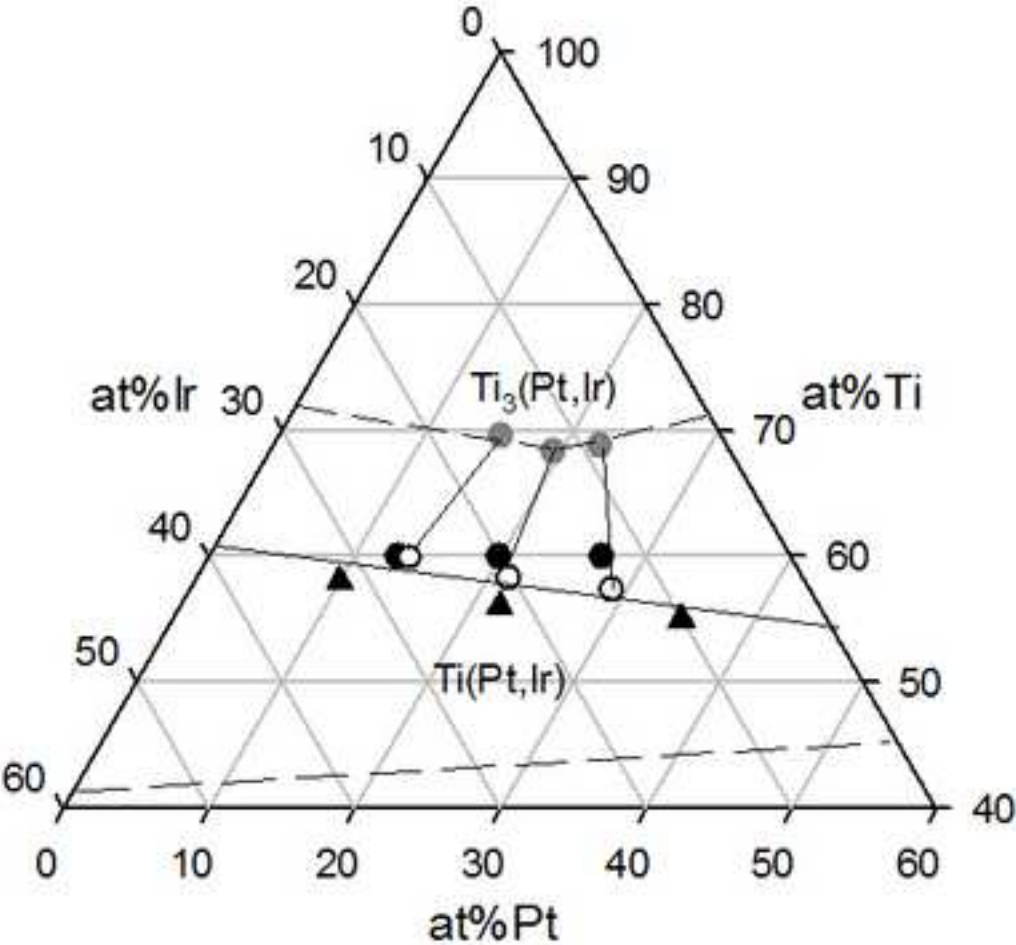
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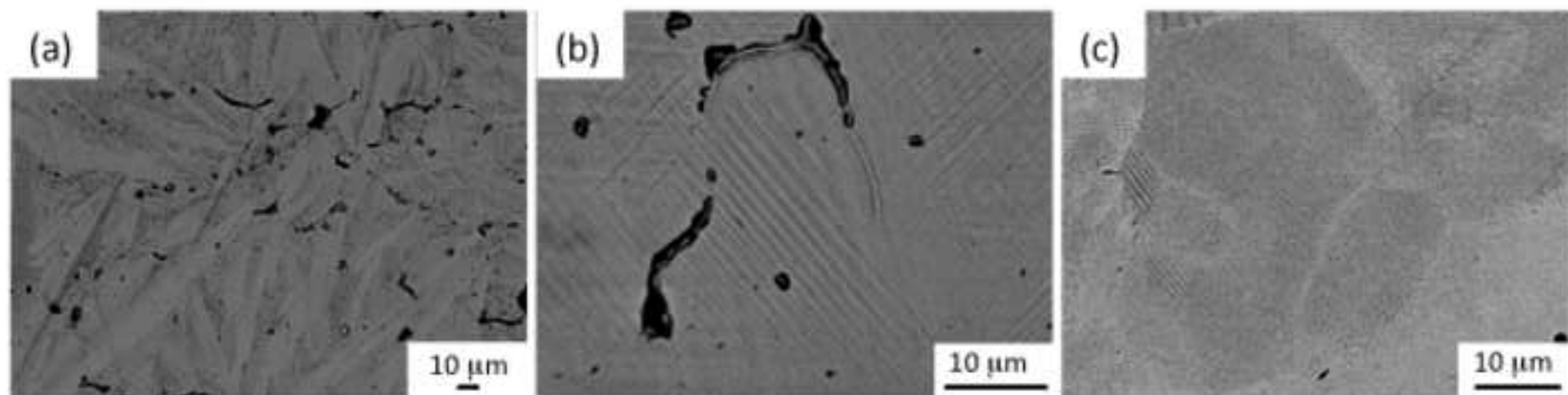
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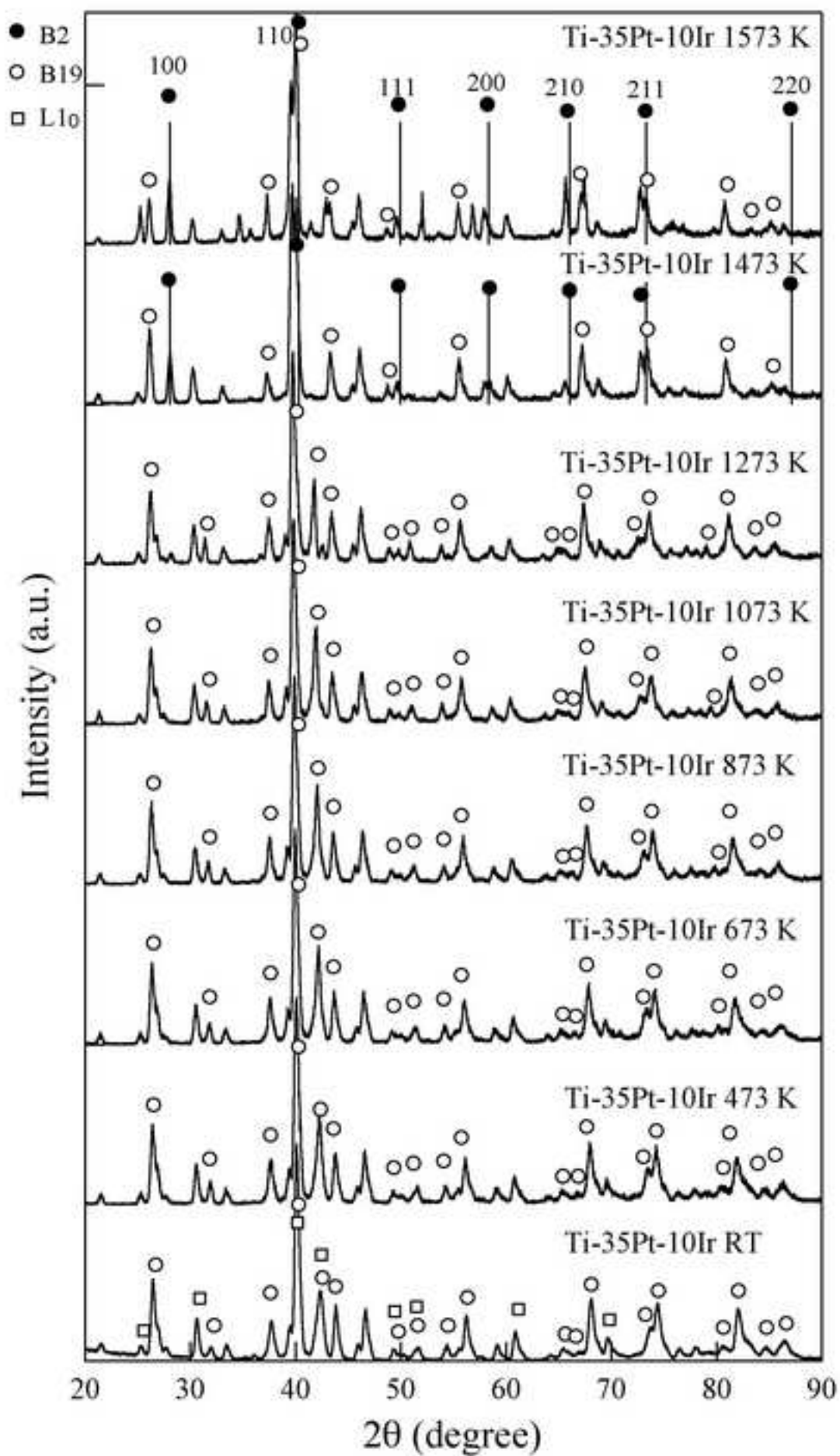


Figure6  
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