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Formation of layer-shaped pores in TiC-Fe cermet by combustion synthesis ①

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[Abstract] To study the formation of layer-shaped pores in TiC-Fe cermet, two Ti-C-Fe powder compacts containing Ti powders with two size ranges (< 44μm and 135~ 154μm) respectively were ignited in a special ignition mode. The combustion temperatures of the reactions were measured, the phase constituents of the combustion-synthesized products were inspected by X-ray diffractometry (XRD), and the structures of the products were observed with scanning electron microscope (SEM). In the case of the finer Ti powder used, TiC-Fe cermet and pore rank in an alternately laminar shape, and the shape of the pore is the same as that of the combustion wavefront, implying that the layer-shaped pore results from a gather of the retained gas into the combustion wavefront. While in the case of the coarser Ti powder used, the lower combustion temperature causes the gather of the retained gas to be difficult, the pore being present in an arbitrary shape and distributing randomly.

[Key words] layer-shaped pores; TiC-Fe; combustion synthesis
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1 INTRODUCTION

The combustion synthesis has been successfully used for fabricating various compounds and composites, such as TiC[1,2], TiC-Ti[3], TiC-Al[4~6], TiC-Al-Cu[7] and TiC-Fe[8~15].

For the combustion synthesis of TiC-Fe cermet, the Ti-C-Fe powder mixture is commonly pressed uniaxially as a cylindrical compact, and the compact is then ignited with an incandescent graphite flat above the top surface of the compact[10~13]. It has been found that when the Ti powder is coarser the pores in the synthesized product are arbitrary shape and randomly distribute[11,13], whereas when the Ti powder is finer the product is an alternately laminar structure of TiC-Fe cermet and pores, and the layer-shaped pores are vertical to the axis of the compact[10]. Thus for the formation of the layer-shaped pores, possible two factors should be considered. One is an existence of residual tensile stress parallel to the axis of the compact, this is helpful to a formation of layer-shaped pores vertical to the axis of the compact. The other is the plane-shaped combustion wave propagating along a direction of the axis of the compact, this is beneficial to a formation of layer-shaped pores with the same shape as that of the combustion wavefront. However, this mode of the ignition at the top surface of the compact causes these two possible factors to be confused with each other.

In this paper, a special mode of ignition is used to separate these two possible factors with each other, and two size ranges of Ti powder are used to study the effects of the combustion temperature on the formation of the layer-shaped pores.

2 EXPERIMENTAL

Two cylinder compacts (d14 mm × 18 mm) with a relative density of about 60% were uniaxially pressed in a steel die, one of which containing 56% titanium powders (< 44 μm), 14% carbon black (0.033 ~ 0.079 μm) and 30% iron powders (135 ~ 154 μm), and the other containing 56% titanium powders (135 ~ 154 μm), 14% carbon black (0.033 ~ 0.079 μm) and 30% iron powders (135 ~ 154 μm). A small hole (d 2 mm × 9 mm) was drilled at the center of the compact bottom, and a junction of W-3%Re—W-25%Re thermocouple with a wire (d 0.1 mm) was inserted into the hole, with the other end being linked up an x-y recorder. The cylindrical compact was horizontally placed in a reaction chamber filled with 0.1 MPa argon, and then ignited with an incandescent graphite flat which was 2 mm above the compact and parallel to the axis of the compact, as shown in Fig.1. Thus a plane-shaped combustion wave was parallel to the axis of the compact and self-propagated through the compact, meanwhile, a temperature-time curve of the combustion reaction was recorded by the recorder, and the phase constituents of the combustion-synthesized products were inspected by XRD. The combustion-synthesized samples were longitudinally cut and prepared as metallogra-

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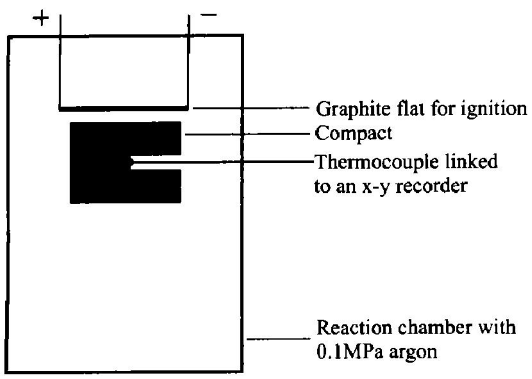

Fig. 1 Schematic diagram of experimental apparatus for combustion synthesis

phic specimens, and the structures were then observed with SEM.

3 RESULTS

Fig. 2 shows the measured temperature-time curves for the compact containing the finer Ti powder (Fig. 2(a)) and for the compact containing the coarser Ti powder (Fig. 2(b)), and it can be seen that the combustion temperature of the former (1 996 ℃) is much higher than that of the latter (1650 ℃).

The measured XRD patterns indicate that, as shown in Fig. 3, the synthesized product of the compact containing the finer Ti powder is composed of TiC and α-Fe, this implies a complete reaction,

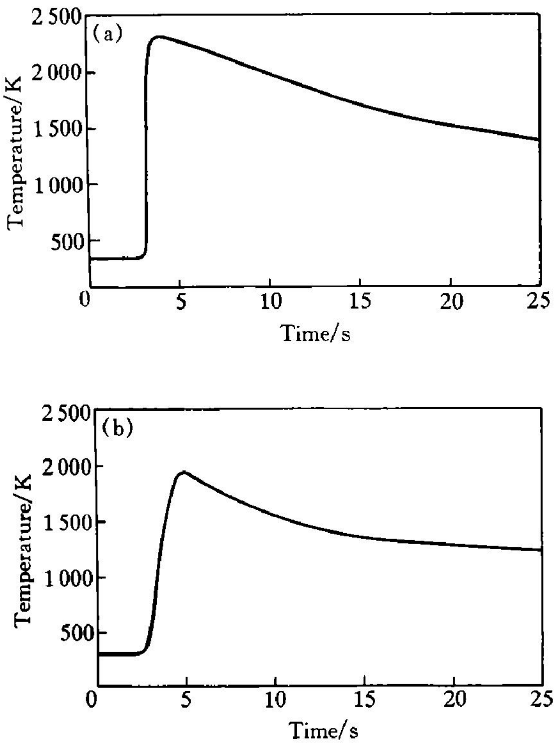

Fig. 2 Temperature-time curves of combustion reaction

(a)—For compact containing finer Ti powder;
(b)—For compact containing coarser Ti powder

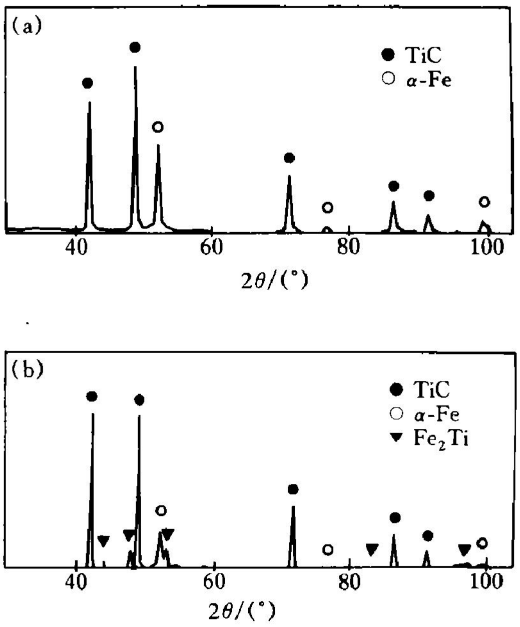

Fig. 3 XRD patterns of combustion-synthesized product

(a)—For compact containing finer Ti powder;
(b)—For compact containing coarser Ti powder

while that of the compact containing the coarser Ti powder consists of TiC, α-Fe and a small amount of Fe_2Ti, this means an incomplete reaction.

Fig. 4 shows the SEM photographs of the combustion-synthesized product of the compact containing the finer Ti powder. As shown in Fig. 4(a), the product is an alternately laminar structure of TiC-Fe cermet and pores, and the layer-shaped pores are parallel to the axis of the compact. The microstructure of the TiC-Fe cermet is shown in Fig. 4(b), the TiC particles have an average diameter of about 4.5 μm.

The SEM photographs of the combustion-synthesized product of the compact containing the coarser Ti powder are shown in Fig. 5. Although the structure consists of TiC-Fe cermet and pores too, as shown in Fig. 5(a), the pores are arbitrary in shape and randomly distribute. In addition, there are some incomplete reaction regions in the combustion-synthesized product, i. e. the white regions in Fig. 5(a). The microstructure of the TiC-Fe cermet is shown in Fig. 5(b), the average diameter (about 3.0 μm) of the TiC particles is smaller than that for the case of the finer Ti powder.

4 DISCUSSION

In the case of the finer Ti powder used, the layer-shaped pores vertical to the axis of the compact are changed into that parallel to the axis with changing the mode of the ignition. This result indicates that the formation of the layered pores is principally related to the combustion wave rather than the residual tensile stress. By rapid-speed motion picture records

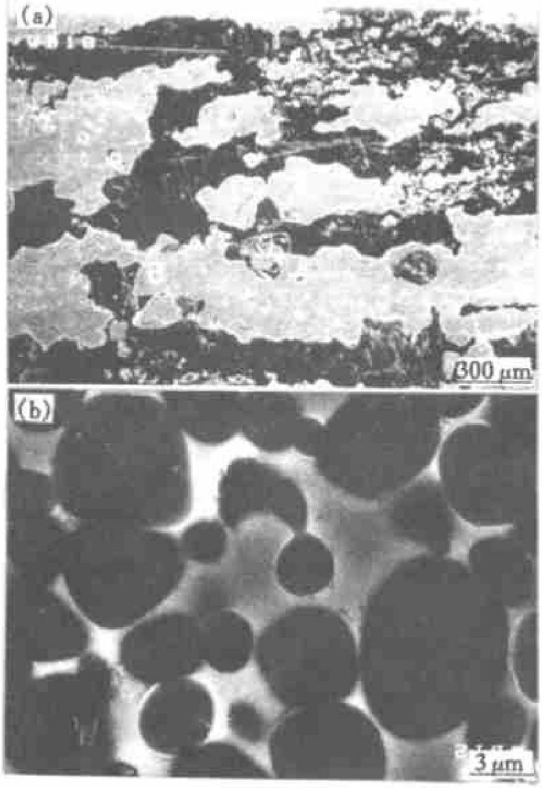

Fig. 4 SEM photographs of combustion-synthesized product of compact containing finer Ti powder
(a)—Macrostructure; (b)—Microstructure

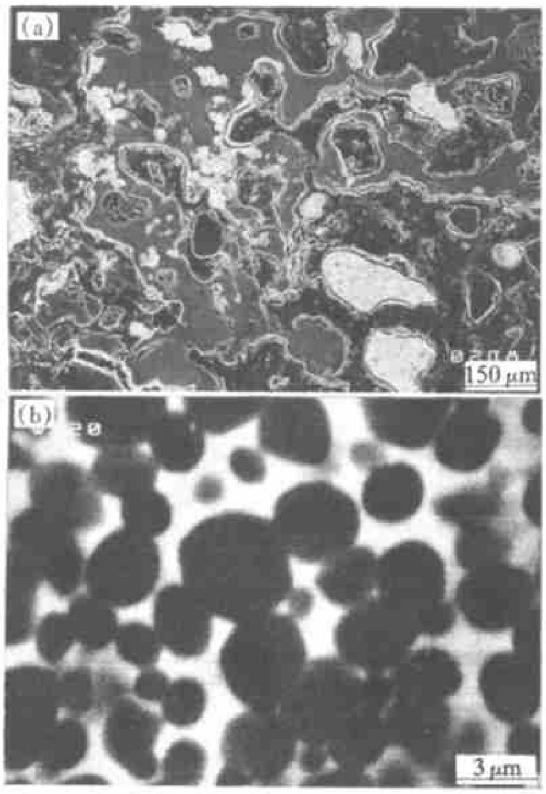

Fig. 5 SEM photographs of combustion-synthesized product of compact containing coarser Ti powder
(a)—Macrostructure; (b)—Microstructure

of the combustion of Ti + C, Holt and Munir[2] have proved evolution of a substantial amount of gas. A portion of the evolved gas escapes from the sample, but the other retains within the sample due to a high combustion rate and tends to get together so as to reduce the free energy of the system. A recent investigation result[16] obtained by means of a combustion front quenching method showed that the combustion reactions in the compact containing the finer Ti powder could be described with a dual-solution-precipitation model. Both the reactions taking place respectively in the Ti and Fe particles proceeded by the solution-precipitation mechanism, and the reacting Ti and Fe droplets fused with each other finally in a plane-shaped wavefront. In the case of the finer Ti powder used, a higher fluidity of the product in the wavefront, due to the higher temperature, is helpful to the fusing, and also helpful to the moving and getting together of the residual gas so as to form the layered pores with a same shape as that of the wavefront. The SEM photograph shown in Fig. 6(a) shows the fusing of the Ti and Fe droplets as well as the forming of the layered pores in the wavefront. An alternately laminar structure of $TiC-Al_2O_3$ ceramic and pores was also observed by Bowen and Derby[17], and the heat flow was considered to be in the direction perpendicular to the laminar structure.

While in the case of the coarser Ti powder used, no layer-shaped pore forms although the mode of the ignition is the same as that for the finer Ti powder

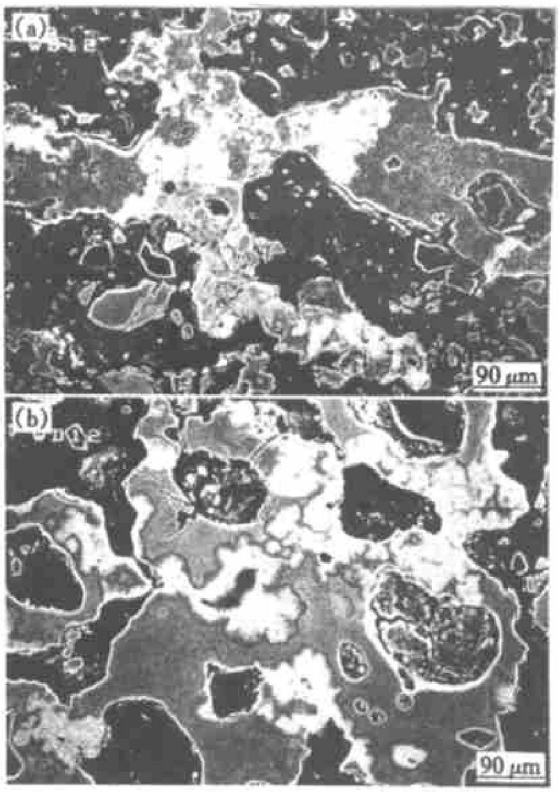

Fig. 6 SEM photographs at combustion wavefront in quenched sample
(a)—Compact containing finer Ti powder;
(b)—Compact containing coarser Ti powder

used. This indicates that the formation of the layered pores is also related to the combustion temperature. The combustion reactions in the compact containing the coarser Ti powder could be described with a ternary-reaction-diffusion/solution-precipitation model[11]. The reactions taking place in the Ti and Fe particles were proceeded respectively by the ternary-reaction-diffusion mechanism and the solution-precipitation mechanism, and the reacting Ti and Fe droplets fused with each other finally in a plane-shaped wavefront. However, the coarser Ti powder causes an incomplete combustion, a lower combustion temperature and a lower fluidity of the product in the wavefront. This is not benefit to the fusing, the moving and the getting together of the residual gas so as to form the arbitrarily shaped and randomly distributed pores. The SEM photograph shown in Fig. 6(b) shows the fusing of the Ti and Fe droplets as well as the forming of the pores in the wavefront.

5 CONCLUSIONS

1) By changing the mode of the ignition, it is proved that in the case of the finer Ti powder used, the formation of the layered pores mainly results from getting together of the evolved residual gas into the combustion wavefront.

2) While in the case of the coarser Ti powder used, the lower combustion temperature is not benefit to getting together of the residual gas so that no layered pore forms.

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