Effects of technological parameters on microstructures and properties of in situ TiC_p/ Fe composites [①]

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Abstract : The effects of the reactive temperature , time and the cooling rate of an Fe- Ti- C alloy melt on the microstructures and mechanical properties of in situ TiC_p/ Fe composites were investigated . The results show that the hardness and impact toughness of the prepared composites increase with increasing the reactive temperature , because more and finer TiC particles are formed in the higher temperature melt . However , after the TiC synthesis reaction in the melt completed , the impact toughness of the composites will decrease if the melt reactive time is further prolonged , owing to the coarsening of the formed TiC particles . Under the present experimental condition , the cooling rate of the melt containing dispersions has little influence on the number , size and distribution of the particles in the composites .

Key words : in situ reaction ; microstructure ; property ; TiC_p/ Fe composites   **Document code : A**

1   INTRODUCTION

TiC is widely used as in situ reinforcing particle in metal matrix composites[1~5] , because it has not only high melting point , hardness , elastic modulus and excellent corrosion resistance , but also more negative standard Gibbs formation free energy . In recent studies[6~9] , however , the raw materials used for the synthesis of TiC particles generally were highly pure titanium and carbon powder , and most work was mainly focused on aluminum matrix composites . In order to prepare in situ TiC particles reinforced ferrous matrix composites (TiC_p/ Fe) by using low cost ferrotitanium alloy and cast pig iron , a novel technique called reactive casting[10,11] was early developed by the authors .

The developed technique above includes the following two processes : one is the isothermal reaction process of a molten Fe- Ti- C alloy to promote the formation of TiC , in which the melt reactive temperature and time basically determine the volume fraction and the size of the TiC particles formed in the melt ; the other is subsequent casting process of the melt containing the formed dispersions for getting a final shape component , in which the cooling rate of the melt may influence the particles distribution in the metal matrix .

Therefore , it is very important to investigate the effects of above technique parameters on the microstructures and mechanical properties of the prepared composites .

2   EXPERIMENTAL PROCEDURE

The main raw materials used in present experiment were ferrotitanium alloy and cast pig iron , whose chemical compositions and usage are listed in Table 1 . In addition , A3 steel , 75 %Si- Fe and 65 % Mn- Fe were used to adjust the alloy composition . A Fe- 4 .0 % Ti- 1 .5 %C- 2 .5 %Si- 0 .4 % Mn master alloy , which was optimized in a specialized article[12] , was prepared respectively in a vacuum or common air induction furnace . In the common induction furnace , a self- made covering flux was applied to protect the melt from oxidization . When the melt reactive time was constant (5 min) , the effects of the melt reactive temperature (1 500 , 1 550 , 1 600 , and 1 650 ℃ respectively) on the microstructures and mechanical properties of the prepared composites were studied systematically . In return , in the vacuum induction furnace , when the melt was maintained at 1 600 ℃ , the effects of the melt reactive time (0 , 5 , 10 , 15 , and 20 min) on the microstructures and properties of the composites were further studied . As described in the earlier work[12] , the microstructures and properties of the prepared composites were examined from the impact samples with a gauge of 20 mm × 20 mm × 110 mm , which were cast in a resin sand mould after the isothermal reaction process of the melt . Additionally , after the melt was treated at 1 600 ℃ for 10 min , it was cast into a stepped specimen with a width of 50 mm and wall thickness of 5 , 10 , 25 and 40 mm , respectively . The effect of the cooling rate of the melt

on the microstructures of the composites was studied by examining the different cross sectional microstructures of the stepped specimen.

3　RESULTS AND DISCUSSION

3.1　Influence of reactive temperature

　　When the melt reactive temperature was 1 500 ℃, as added ferrotitanium alloys with a melting point of about 1 530 ℃ almost could not melt, the melt only contained a few free Ti atoms to react with C atoms. As a result, there were few TiC particles in the obtained specimen, whereas a large number of block-like unmelted ferrotitanium alloys were visible on the fracture surface of the specimen. When the reactive temperature was up to 1 550 ℃, the amount of free Ti atoms in the melt was significantly increased because of the melting of most ferrotitanium alloys, resulting in the formation of a lot of TiC particles in the melt. During subsequent solidification process of the melt, the formed TiC particles acted as the heterogeneous nuclei of austenite crystals to refine grains, while the unreacted C atoms were preferentially segregated to the grain boundaries in the form of dot-like graphite, as shown in Fig.1(a). As the reactive temperature was further increased, the amount of free Ti atoms in the melt was also increased, and so did the diffusing rate of both Ti and C atoms and their combining probability in the melt. As a result, the TiC particles in the solidified specimen became large in number and finer, while the size of the austenite grains and the amount of the graphite at grain boundaries were all decreased. These changes can be clearly seen by comparing the microstructures shown in Fig.1(a) with that shown in Fig.1(b). When the melt was maintained at a higher temperature (e.g. 1 650 ℃), a large number of TiC particles with much finer size were formed in the solidified specimen, and they exhibited more homogeneous distribution, as shown in Fig.1(c). Additionally, it should be noted from Fig.1(c) that the graphite phase as appeared in Fig.1(a) and Fig.1(b) has disappeared, which implies that the synthesis of TiC in the melt has basically completed.

　　Fig.2 illustrates the effects of the reactive temperature on the microstructures and mechanical properties of the composites. It can be seen that, under the present experimental condition, when the reactive temperature is higher than 1 550 ℃, TiC particles begin to form in large quantities. Furthermore, with increasing the reactive temperatures, the number of the particles is also increased, whereas the average size of the particles is decreased (Fig.2(a)), which leads to increase in both the hardness and impact toughness of the prepared composites (Fig.2(b)). Consequently, as long as the practical conditions are acceptable, properly increasing the melt reactive temperature is favorable to fabricate the composites with finer microstructure and better properties.

3.2　Influence of reactive time

　　Fig.3 shows the solidified microstructures of the melt treated at 1 600 ℃ for different time periods. It can be seen that the melt reactive time has an obvious influence on the microstructures of the composites. When the melt was not through the isothermal reactive process, namely, its reactive time was 0 min, as Ti and C atoms in the melt had not chance to

Table 1　Chemical compositions and usage of main raw materials

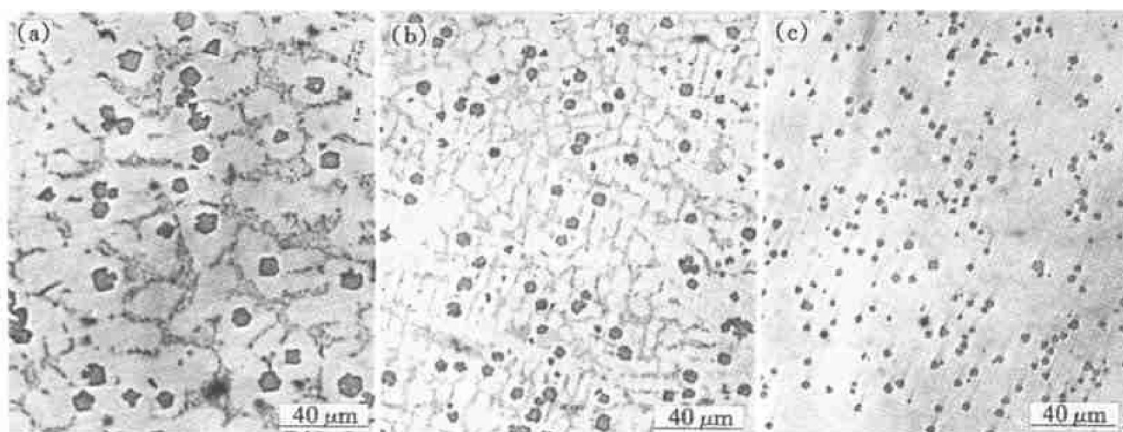| Material	Chemical composition	Usage
Ferrotitanium	40.56 %Ti, 0.084 %C, 0.047 %P, 0.022 %S, Bal.Fe	To provide Ti atoms
Pig iron	4.13 %C, 1.21 %Si, 0.16 %Mn, P < 0.03 %, S < 0.03 %, Bal.Fe	To provide C atoms; matrix of composites

Fig.1　Cast microstructures of melt at different temperatures for 5 min
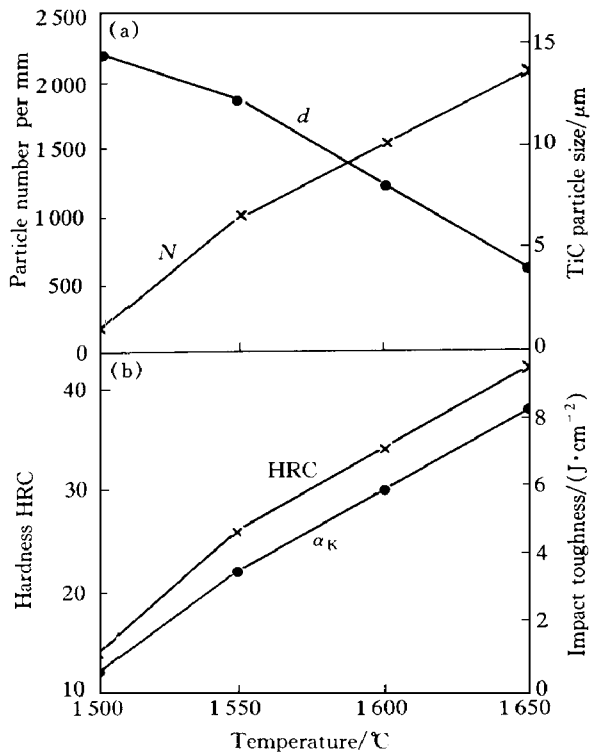(a) —1 550 ℃; (b) —1 600 ℃; (c) —1 650 ℃

Fig .2   Effects of reactive temperature on microstructures (a) and properties (b) of composites

synthesis into TiC, only a few TiC particles were formed in the solidified specimen. However, a large amount of plank-like cementites were precipitated due to the element Ti action of strongly promoting the formation of cementite during solidification, as shown in Fig .3(a) . After the melt was isothermally treated for 10 min, its solidified specimen contained a large number of fine TiC particles with a homogeneous distribution instead of the above mentioned cementite, as shown in Fig .3(b) . This further indicates that the isothermal treatment for the melt is essential to obtain desirable TiC particles in the composites . However, if the reactive time is further prolonged after the TiC

synthesis process completed in the melt, the formed TiC particles will obviously grow . For example, the size of the particles in Fig .3(c) (reactive time 20 min) is larger than that in Fig .3(b) (reactive time 10 min) , and the former has less number of the particles than the latter . Accordingly, the number and size of particles in the composites can be effectively controlled by adjusting the reactive time of the melt .

Fig .4 shows the effects of the reactive time on the microstructures and mechanical properties of the composites . It can be seen that there exists an optimum reactive time, which is about 10 min in the present study . When the reactive time was shorter than 10 min, with the prolongation of the time, the average number of the TiC particles was significantly increased, while their average size was also increased slightly, owing to the continuous formation of the particles in the melt . However, because the TiC synthesis reaction in the melt was not completed during this period, some graphite was precipitated at the grain boundaries, as shown in Fig .1 (b) . The existence of graphite phase caused the prepared composites to have lower hardness and impact toughness . When the reactive time was longer than 10 min, although the hardness of the composites had a little increase because of the disappearing of the graphite phase, its toughness was obviously decreased due to the coarsening of the particles . Only when the reactive time was 10 min, both the hardness and toughness of the composites were the highest, because the largest number of and the finest TiC particles were formed in the composites, as shown in Fig .4 . In addition, it can be noted that, by comparing Fig .2(a) with Fig .4(a) , the influencing rule of the reactive temperature on the number and size of the particles in the microstructure is contrary to that of the reactive time . This fact implies that there is a matching relationship between the reactive temperature and the reactive time . In general, under the condition of ensuring the completion of the TiC forming reaction in the melt, a selection of higher reactive temperature and shorter reactive time
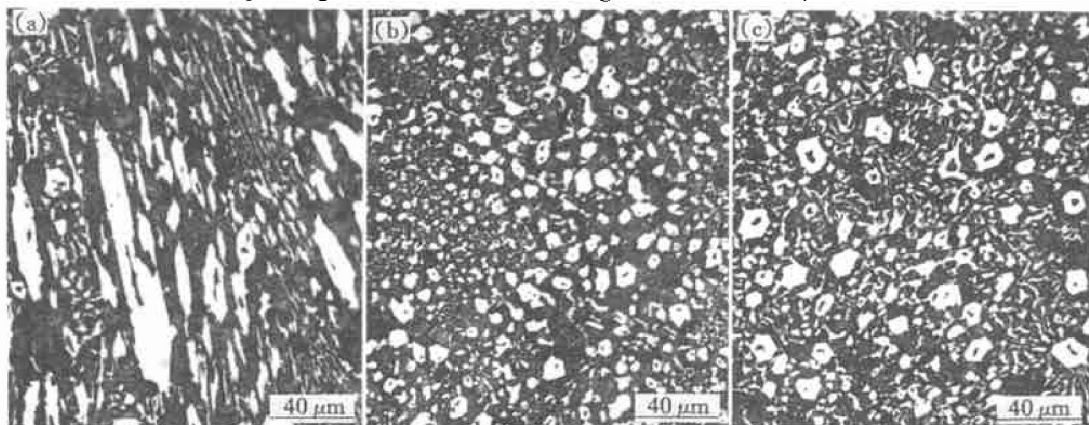

Fig .3   Cast microstructures of melt reacted at 1 600 ℃ for different time periods
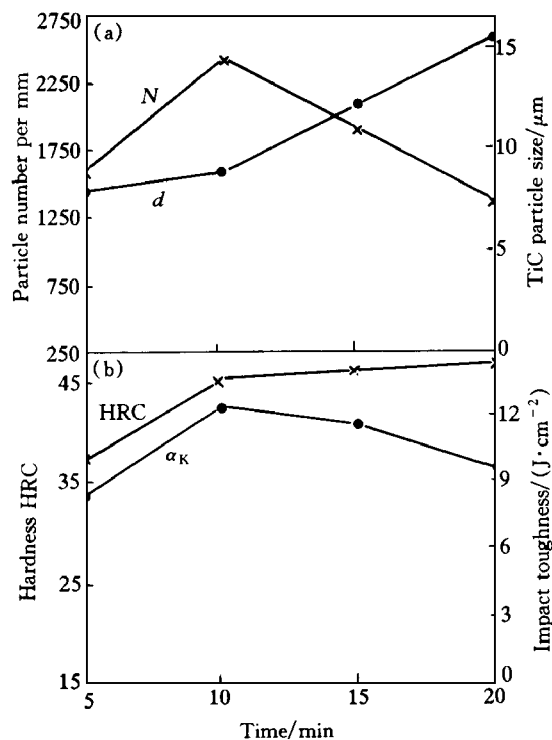(a) —0 min; (b) —10 min; (c) —20 min

Fig .4　Effects of reactive time on micro-
structures (a) and properties (b) of composites

is favorable to obtain more and finer TiC particles in
the composites .

3 .3　Influence of cooling rate

　　Fig .5 shows three cross sectional microstructures
in a stepped specimen with different wall thickness .
As mentioned previously , the particles in the compos-
ites were formed by the reaction in the melt . There-
fore , when the reactive temperature and time were
constant , the particles formed in the melt should have
constant number , size and shape . During following
solidification of the melt , as long as castings have lit-
tle difference in wall thickness , in other words , if the
cooling rates of the castings are almost same , the
number , size and shape of the particles distributed in
the castings will have little changes . This result can
be demonstrated by Fig .5(a) (wall thickness 40 mm)
and Fig .5(b) (wall thickness 25 mm) . However , a
great variation in cooling rate can result in some
changes in the solidified microstructure . For example ,
the size of the particles in Fig .5(a) is a little larger
than that in Fig .5(c) (wall thickness 5 mm) . More-
over , some finer TiC particles were further precipitat-
ed in the former specimen , whereas a little web-like
cementite was formed in the latter specimen . After
the melt being isothermally , reactively treated , a few
unreacted Ti and C atoms were remained in the melt .
When the wall thickness of a casting was larger , the
poured melt could stay for a longer time due to its
slower cooling rate . Therefore , those remained Ti and
C atoms could further combine into finer TiC parti-
cles , some of which directly maintained in the subse-
quent solidified specimen , while the others coalesced
on the initial particles in all directions to cause the
particles growing , as shown in Fig .5(a) . However ,
during the solidification process of a thin wall casting ,
the Ti and C atoms remained in the melt did not have
time to combine into TiC , but they could solubilized
in the primary austenite grains . As the solidification
proceeded , the austenite grains grew and extracted Ti
and C atoms into the remained liquid phase at the
grain boundaries . When Ti content in the liquid phase
was increased to certain extent , the liquid phase was
transformed into the cementite according to Fe- Fe_3C
quasi-steady system . As a result , the generated ce-
mentite distributed at grain boundaries , whereas TiC
particles basically maintained their initial size and
shape in the melt , as shown in Fig .5(c) . In general ,
under the present experimental condition , the cooling
rate of the melt has little influence on the
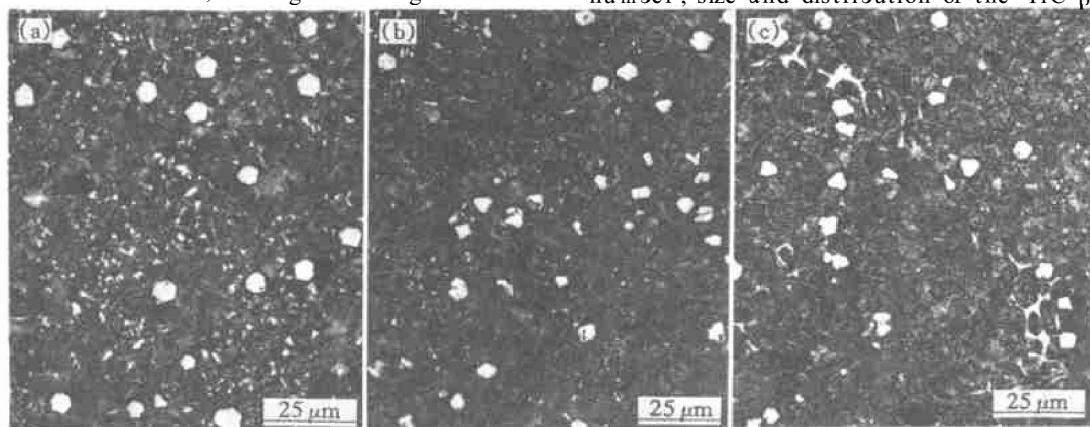number , size and distribution of the TiC particles in

Fig .5　Microstructures with different wall thicknesses in stepped specimen
(a) —40 mm ; (b) —25 mm ; (c) —5 mm

the composites .

4 CONCLUSIONS

1) Under the present experimental condition , when the reactive temperature of the melt is higher than 1 550 ℃ , a large number of TiC particles are formed in the melt ; and with increasing the reactive temperature , the number of the particles is increased , whereas their average size is decreased . As a result , both the hardness and the impact toughness of the prepared composites are improved with increasing the melt reactive temperature .

2) As the melt reactive time is prolonged , TiC particles are continuously formed in the melt . However , after the TiC synthesis reaction being completed in the melt , if the reactive time is further prolonged , the impact toughness of the prepared composites will decrease because of the coarsening of the formed particles . Therefore , there is an optimum reactive time for a certain composition alloy melt .

3) There exists a matching relationship between the melt reactive temperature and time . Under the condition of ensuring the completion of the TiC forming reaction in the melt , a selection of higher reactive temperature and shorter reactive time is favourable to fabricate the composites with finer microstructure and higher mechanical properties .

4) As long as the TiC synthesis reaction in the melt is basically completed , the cooling rate of the melt has little influence on the number , size and distribution of the particles in the composites under the present experimental condition .

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