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MICROSTRUCTURE AT THE INTERFACE OF TITANIUM CARBIDE AND NICKEL ALUMINIDES *

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Microstructure at the interface of titanium carbide and nickel aluminides in the samples obtained by infiltration of molten Ni_3Al alloy has studied by a scanning electron microscopy (SEM) and an analytical transmission electron microscopy (ATEM) with an energy dispersive spectrometer (EDS). It is found that the morphology at the interfaces between hard phase skeleton of $TiC_{0.7}$ and metallic phases depends on the ratio of Ti/C in carbide. Some periodic zigzag fringes are observed at a smooth interface between metallic phase and carbides in the sample of $Ni_3Al/TiC_{0.7}$. The results of analysis using EDS show that Ti in $TiC_{0.7}$ carbide is easier than that in $TiC_{0.9}$ to dissolve into the molten alloy during solid-liquid reaction. The formation of this periodic zigzag fringe,which may be a growth zone of a new Ti-Ni-Al phase,in the interface of $TiC_{0.7}/Ni_3Al$ would occur during the initial stage of solidification.

Keywords: titanium carbide, nickel aluminides, interface, microstructure
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I. INTRODUCTION

Recently, ordered intermetallic compounds, particularly the aluminides of nickel, titanium, iron and niobium have been under intensive investigation as advanced load-bearing materials for high temperatures, with special emphasis on aerospace applications. The main problem with these high strength phases is extreme brittleness at or near ambient temperature. Possible solutions appeared to be alloying with certain elements in order to obtain a two-phase or multiphase material with an enhanced plastic deformability even at ambient temperature. The introduction of reinforcer such as oxides or carbides into an intermetallic matrix may, however, produce improved high temperature strength, and creep resistance at elevated temperatures and low temperature toughness compared with single-phase materials. Studies of such composite materials (e.g. SiC, Si_3N_4, TiC, TiB_2 and Al_2O_3, embedded in Al-Ni or Al alloys) have been widely reported during the past several years.[1]

Since the durability and mechanical performance of composites are influenced to a great extent by the properties of the interface formed between the matrix and the reinforcer,the control of interfacial interactions or reactions is essential for development and manufacturing of such materials.[2]

In the present studies we show the system of Ni_3Al with TiC_x(x=0.7, 0.9). TiC_x with different C/Ti ratio will be used to optimize the adhesion and phase evolution in the composite materials fabricated by the metallic melt impregnation method. The purpose of the present study are to characterize the interfacial microstructure of metal-carbide composites using a scanning electron microscopy (SEM) and an analytical transmission electron microscopy (ATEM) with an energy dispersive spectrometer (EDS).

Titanium carbide exists over a wide range of composition (0.5 <C/Ti< 1), its chemical, physical and mechanical properties are composition dependent. The non-stoichiometric titanium carbide was obtained by mixing stoichiometric TiC powder with Ti metal powder (2–10 μm) in appropriate ratio prior to sintering. The mixed powders were dry-pressed and then sintered in vacuum at 1800°C for 2 hours.

Various manufacturing methods and raw material forms are used for production of metal-ceramic composites: solid phase sintering, liquid phase sintering or impregnation of porous ceramic in molten metals. In

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solid state sintering it is very difficult to achieve high-density composite. While the method based on liquid metal impregnation into porous ceramics can yield densities as high as 99.5% of the theoretical value. The liquid impregnation techniques benefit from a low viscosity of the molten metal, providing an economical and near net-shape production capability.

In order to investigate the interfacial microstructures of Ni-Al/TiC_x in composites after infiltration, the samples of composites were prepared by the following method: the carbide of TiC_x was dipped into and coated by molten Ni-Al alloy in a vacuum of 10^{-3} Pa. TEM specimens were prepared according to the cross-section method. The sample was cut in two parts which were glued together face to face, slice of 0.2-0.3 mm thick were cut from the glued parts, and then the slices were mechanically polished and argon milled.

Microstructure observations and chemical microanalysis were performed with a CAMBRIDG S-360 scanning electron microscopy and a JEOL2000FX transmission electron microscope both equipped with an energy dispersive spectrometer (EDS).

Ⅱ. RESULTS AND DISCUSSION

As shown in Fig.1, the areas of the intergranular Ni_3Al matrix can be considered as a continuous metallic phase (bright areas) embedded in the hard-phase skeleton (dark areas) because the liquid of Ni-Al was soaked into carbide phase more than 1mm depth in the samples obtained by dipping and coating. The hard phase grains consist of $TiC_{0.9}$ and $TiC_{0.7}$ as shown in Fig. 1 (a) and (b). The composition of both phases was confirmed by EDS.

Comparing the microstructures of samples obtained from $TiC_{0.9}$ and $TiC_{0.7}$, respectively, we see the continuity of the liquid of Ni-Al in $Ni_3Al/TiC_{0.7}$ is better than in $Ni_3Al/TiC_{0.9}$ due to the liquid of Ni-Al has better wetting of $TiC_{0.7}$. Furthermore,in $Ni_3Al/TiC_{0.7}$ most of the hard-phase grains show a more obtuse angular shape with smooth surfaces. This suggests that a stronger interfacial reaction occurred in the interface of $Ni_3Al/TiC_{0.7}$ system during infiltration process compared with the $Ni_3Al/TiC_{0.9}$ system. It may be demonstrated that the continuity of metallic phases in the intergranular areas of carbide and the solubility of the carbide phases in metallic phases depends on the ratio of Ti/C in carbide.

Selected electron diffraction patterns of hard-phase grains show cubic NaCl structure that is a characteristic of TiC_x phases shown in Fig. 2 (a) [001],(b) [215] zone. The diffraction patterns of metallic phases at the interface of Ni-Al/TiC_x and in metallic matrix are shown in Fig.2 (c) and (d). The fcc structures of $[11\bar{1}]$ and $[31\bar{3}]$ zons indicate that one of the metallic phases is $AlNi_2Ti$.

EDS analysis indicates that the composition of the Ni_3Al alloy is modified during infiltration, via dissolution of Ti, and the concentration of element Ti decreases from the hard phase boundary to the center of inter-grains in both samples. The data show that the content of Ti (1.5at% or 3.72at%) in the center of intergranular area of $Ni_3Al/TiC_{0.9}$ is much lower than that in $Ni_3Al/TiC_{0.7}$ samples (15at%). The Ti/C ratio dependence of Ti dissolved in Ni-Al alloys can be explained by phase diagram calculation.[3] On the other hand, there was no evidence of Ni or Al solubility in the TiC_x skeleton.[4]

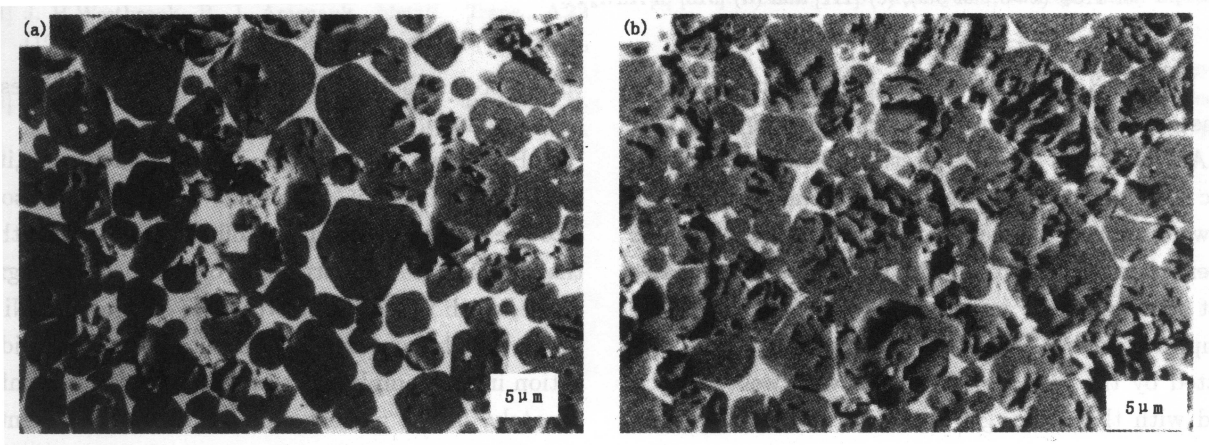


Fig.1. Morphologies in the interface of Ni_3Al/TiC_x by SEM: (a) $Ni_3Al/TiC_{0.7}$, (b) $Ni_3Al/TiC_{0.9}$.

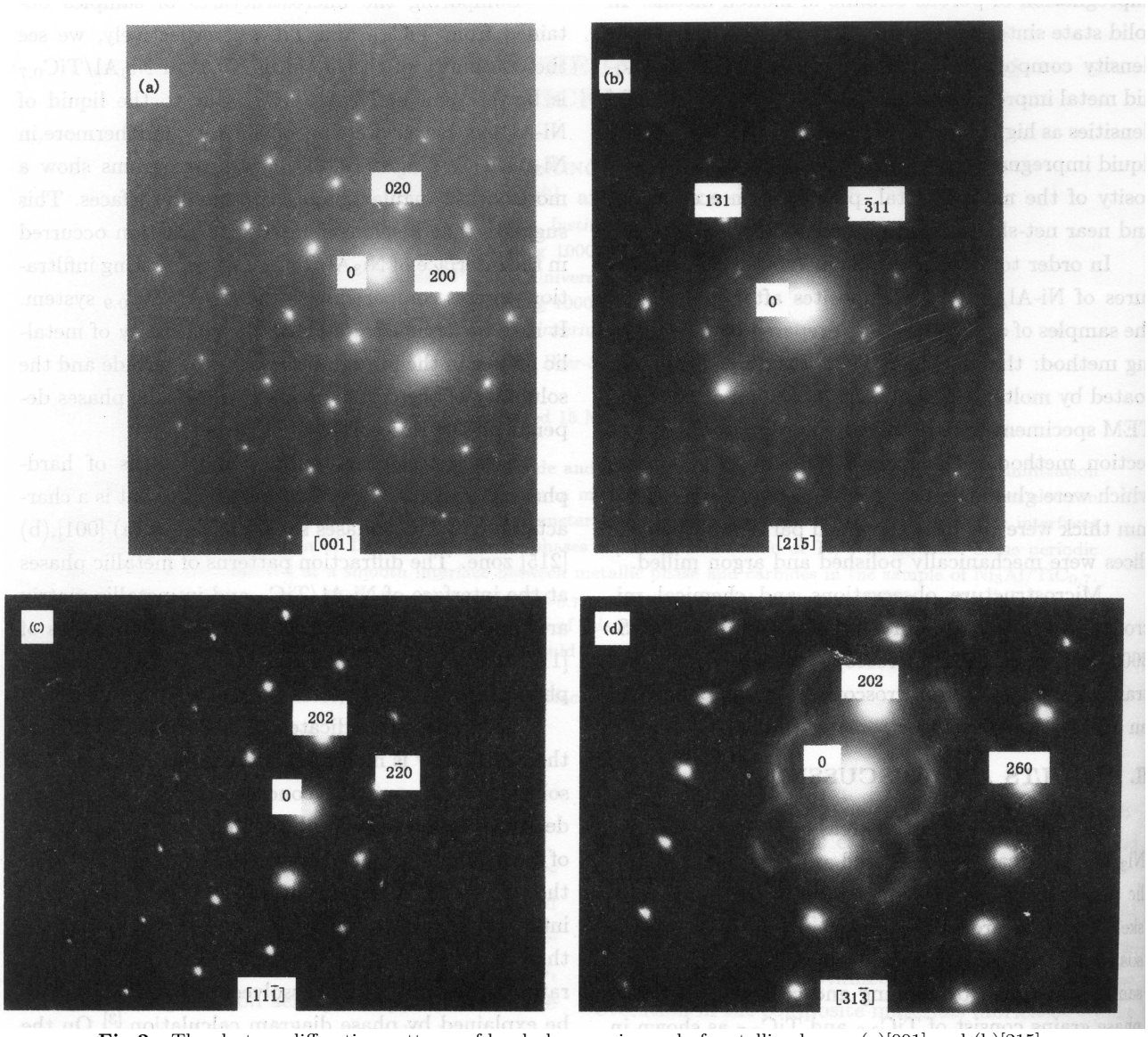


Fig.2. The electron diffraction patterns of hard-phase grains and of metallic phases: (a)[001] and (b)[215] of TiC_x (x=0.7 or 0.9), (c) [$11\bar{1}$] and (d) [$31\bar{3}$] of $AlNi_2Ti$.

Figures 3 (a) and 3(b) show different magnifications of TEM micrographs taken for the interface of $Ni_3Al/TiC_{0.7}$. It is very interesting that some periodic zigzag fringes are observed at a smooth interface between metallic phase and carbides under a suitable observation condition. TEM investigation indicates that these periodic images could not appear in the samples of higher C content,in which the $TiC_{0.9}$ was wetted by the liquid of Ni-Al not very well. Associated with these zigzag fringes, it can be found that some periodic parallel patterns with a 40 nm spacing in the continuous metallic phase areas. The periodic images in the TEM observation can be attributed to the ordered structure that may modulate by phases or orientations of crystal lattices.[5]

By means of the phase diagram calculation,it is clear that Ti in $TiC_{0.7}$ carbide is easier to dissolve into the molten alloy during solid-liquid reaction than that in $TiC_{0.9}$. The formation of this periodic zigzag fringe,which may be a growth zone of a new Ti-Ni-Al phase,would occur during the initial stage of solidification in the samples obtained by infiltration. Unfortunately, in the present study we could not identify the phase or determined its chemical composition in these patterns. Therefore,it is difficult to explain the pattern formation. However, the results of EDS imply

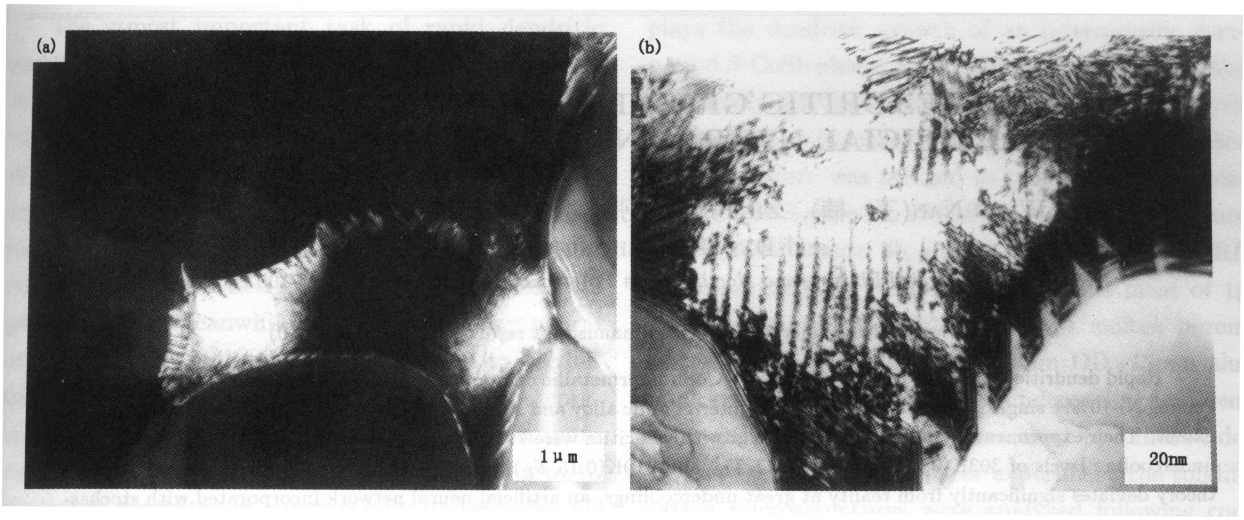


Fig.3. TEM micrographs taken for the interface of $Ni_3Al/TiC_{0.7}$,(a) 1000×. (b)5000×

that formation of these patterns would be caused by a certain amount of Ti dissolved into the alloy during infiltration process. Detailed ATEM work would be necessary to clarify the phenomena. This behavior of interface will improve the strength,creep resistance at elevated temperature and low temperature toughness of the material. The microstructure observation and microanalysis demonstrated the important effect of the C content in the carbide. The beneficial effect of the C content in the carbide contribute to the modification of composite properties. These results are consistent with previous works that have demonstrated the benefits of suitable C content in TiC_x carbide.[1)]

In summary,the system of Ni_3Al with TiC_x (x=0.7, 0.9) shows different behaviors at the interface between the carbide phase and the metallic phase. It is found that the morphology and microstructures at the interfaces between the hard-phase skeleton of TiC_x and the metallic phases depend on the ratio of Ti/C in carbides. The results of EDS and selected area electron diffraction demonstrate the formation of the periodic pattern at the interface of $TiC_{0.7}/Ni_3Al$,and the metallic matrix consists of the solid solutions of Ti in Ni_3Al, which results from dissolution of Ti in $TiC_{0.7}$ carbide during infiltration.

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