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Application of liquid aluminium and $FeO \cdot TiO_2$ powder to the synthesis of composites

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Two methods of *in situ* reinforced phases in aluminium composites were presented in that paper. Based on reaction between ilmenite and aluminium during infiltration (method A) and production of suspension Al_2O_3 and Al-Ti, Al-Fe phases (method B) were synthesised. Presence of intermetallic and ceramic phases were confirmed by XRD and EDS methods.

1. INTRODUCTION

The *in situ* formation of aluminium alloy based composites is preformed using the chemical reaction of some chemical compounds with aluminium in solid state (PM methods) as well as in liquid state (reaction with a solid body)[1-5]. These methods require distributing the reagents in the Al matrix in a way, which allows obtaining a uniform distributing of the newly formed reinforcing phases. In the case of a number of technological solutions, this necessitates proper control of the synthesis process. The synthesis of both ceramic and intermetallic phases in an aluminium matrix is relatively difficult to carry out, since the oxygen, which liberates in the exothermic (aluminothermic) reaction brings about both the formation of Al_2O_3 particles and frothing of reaction products [3,5]. The principal materials used for a reaction with aluminium are the following metal oxides: TiO_2, B_2O_3, NiO, SiO_2, FeO [4]. It seems obvious that the exothermic reaction of aluminium with oxides leads to the formation of Al_2O_3 particles in matrix as well as to enrichment of matrix by a reduced metal, which leads to formation of intermetallic phases. The kinetics of the reaction between oxides and aluminium depends on the degree of reagents' dispersion, their phase and chemical composition, volume fraction and temperature.

The exchange reaction (aluminothermal reaction) between aluminium and ilmenite in this work applied. Ilmenite is titanium iron ore containing 36.8%Fe, 31.6%Ti and 31.6%O (density 4.72 g/cm^3). In high temperature ilmenite reacts with aluminium (exothermal reaction) gives pure Ti and Fe (as an alloy) and Al_2O_3 particles. That reaction courses according the scheme:

$$FeO \cdot TiO_2 + 2Al \rightarrow Al_2O_3 + Fe + Ti \qquad \Delta G = -380 \text{ kJ/mol (at 780 }^{o}\text{C)}$$

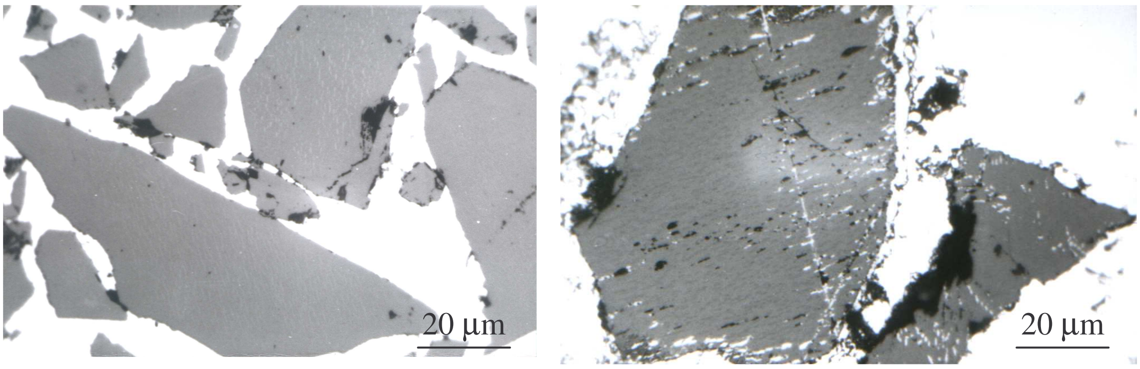
In excess of Al taking participation in reaction, the creation of Al_3Fe and Al_3Ti intermetallic phases became (dissolubility of Fe and Ti in Al below temperature of solidus is 0.04 %Fe and about 0.3%Ti respectively [6]). This reaction can be used to the production of large group of composites, on the intermetallic matrix also.

2. MATERIALS FOR THE RESEARCH

For the formation of a heterophase composite, a $FeO \cdot TiO_2$ ilmenite powder produced by Titania Company of grain size below 70 μm was used as well as A0 grade aluminium of technical purity, produced by the Institute of Non-Ferrous metal in Skawina.
Basis on that results two types of composites have obtained:

3. PRODUCING OF COMPOSITES

The technological conception presented in this paper assumes that the composite formation process will be carried out at two stages. Such a solution was based on the results of a derivatographic study of reaction of Al with $FeO \cdot TiO_2$. Reaction between aluminium and ilmenite begins at a temperature of 710°C and its intensive course is observed at 830°C [8].



(a) (b)

Fig.1: Structure of composite (a) after infiltration of loose ilmenite powder by liquid aluminium under 15 MPa pressure at 640 °C (composite A), (b) after producing of suspension at 700°C (composite B).

Basis on that results two types of composites have obtained:
1) Infiltration of loose powder of ilmenite by liquid aluminium in Degussa press at 15 MPa pressure and at 640°C (temperature has been measured inside of die). In next step, after infiltration the composite held at temperature of 840°C during 1 hour. All procedures have been made in vacuum (composite A).
2) Using technology of production of suspension describing in the work [7], composite suspension containing 30wt.% of ilmenite powders has been produced. Temperature of liquid metal during production of suspension was not high than 700 °C. Composite suspension poured into the ingots. At the second stage the obtained material was subjected to a synthesis in corundum casting powder in argon atmosphere under pressure of 2 MPa at 1000°C/1h.

Such a method of conducting the synthesis ensured a slow course of the aluminothermic reaction, thus minimizing the composite material porosity (composite B). Structures both of composites on that stage of technology are similar (Fig.1)

4. RESEARCH RESULTS AND THEIR ANALYSIS

Assuming that the reaction between Al and ilmenite takes place with a considerable excess of aluminium according to the formula:

$$FeO \cdot TiO_2 + (8+x) \cdot Al \rightarrow Al_2O_3 + Al_3Fe + Al_3Ti + x \cdot Al$$

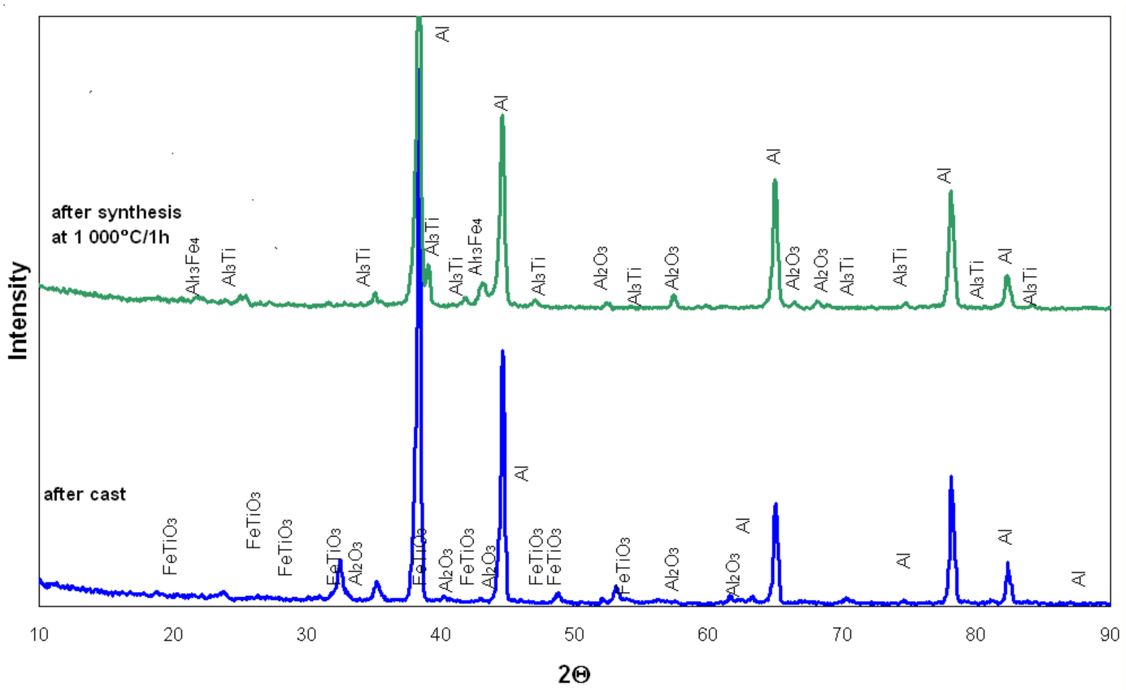
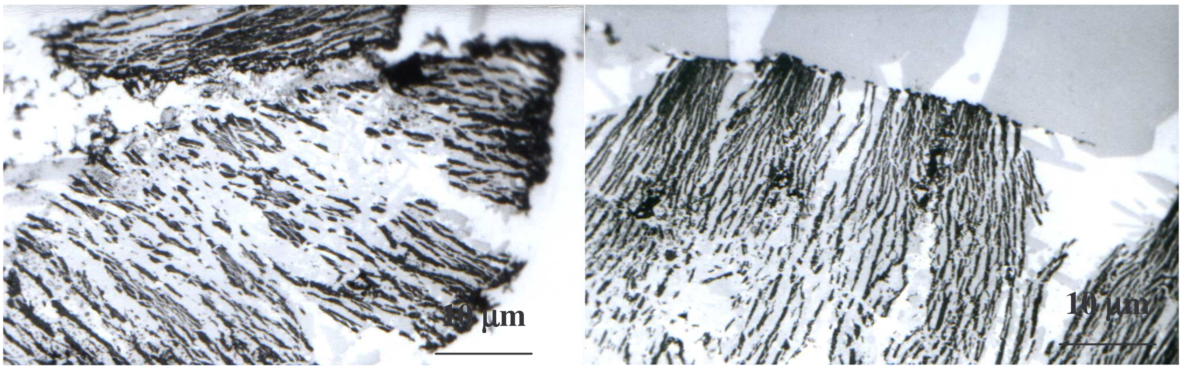


Fig.2. X-ray diagram of Al-FeO·TiO$_2$ system after producing of suspension at 700°C and after synthesise at 1000 °C.

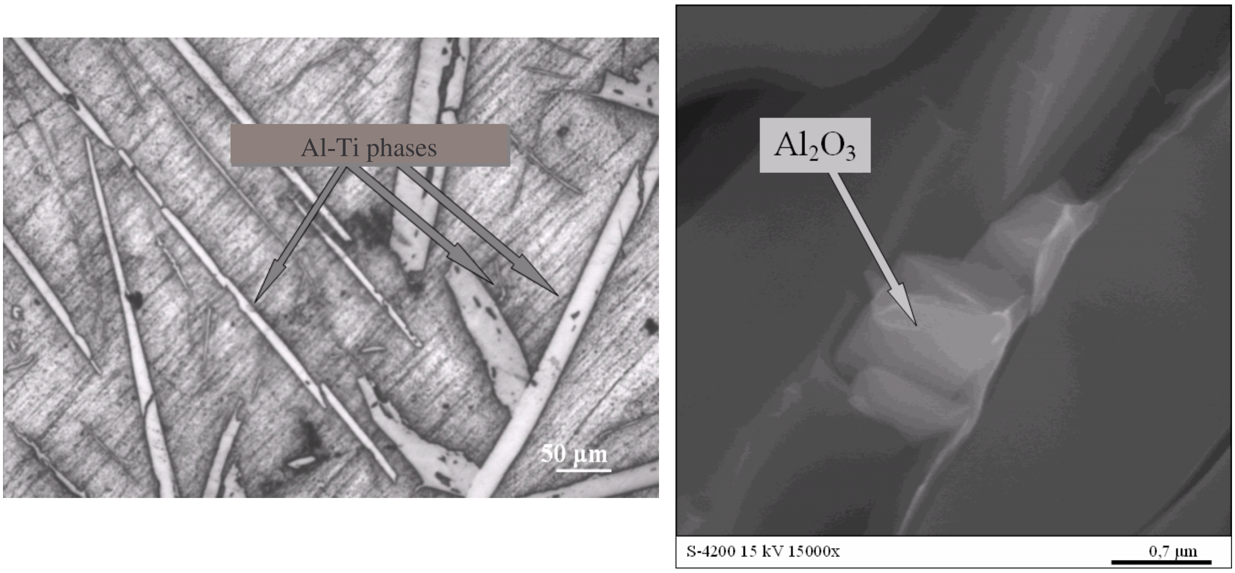
Then, under equilibrium condition, the following phases are formed in the composite (for 30wt.% of introduced ilmenite): 17,6vol.% of Al_3Fe phase, 19,7 vol.% of Al_3Ti and 12,9 vol.% of Al_2O_3. Total volume fraction of reinforced in this composite is about 48%.
The investigation by XRD method for all composites have been confirmed that after synthesis in 840°C (composite A) and 1000°C (composite B), phase constitution of composites is agree with assumption (Fig.2).



(a) (b)

Fig 3. Structure of composite: (a) after synthesis of composite precursor A at 840 ^oC/1h, (b) after synthesis of composite precursor B at 1000^oC/1h

Furthermore, the large crystallites of new phases in aluminium matrix are legible. The microstructure analysis of composites has been made by light microscopy method (LM) and scanning electron microscopy (SEM and EDS methods) confirmed also that changes. The structures observed by LM have been shown on the Fig. 3.



(a) (b)

Fig.4. (a) Multicomponent phases in composites obtained *in situ* after synthesis Al-FeO·TiO_2 composite precursors at 1000 ^oC/1h, (b) Al_2O_3 particle in composites.

After synthesis at 840^oC (composite A) and at 1000^oC (composite B) in the structure of composites are present large particles with lamellar composed structure and size corresponding to primary particles of ilmenite. Furthermore, the large crystallites of new phases in aluminium matrix are legible.

The results of observation of composites (A) and (B) are similar. As mentioned above in the aluminium matrix the presence of intermetallic particles from Al-Fe and Al-Ti systems are stated. The multi components particles size corresponds to the applied ilmenite particles are also found (Fig. 4 a). Predominate components were fine dispersed phases of Al_2O_3 and phases from Al-Fe and Al-Ti systems. Aluminium was only in micro areas.
Analysis by EDS method of multi component particles (Fig. 4 b) let to defined areas pointed to the presence of fine dispersed Al_2O_3 particles and lamellar phases from Al-Ti and Al-Fe systems.

5. CONCLUSION

1. Investigations of structure of composites are confirmed technological concept to the production of composites by *in situ* method using to ilmenite and aluminium. The composites consist of fine dispersed Al_2O_3 particles and intermetallic phases from Al-Ti and Al-Fe systems.
2. Morphology and chemical composition of composites obtained of both methods for this same volume fraction of ilmenite in aluminium matrix are similar.

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