High Porosity Alumina as Matrix Material for Composites of Al-Mg Alloys

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Abstract. The sophisticated industry and technologies require higher and higher assumptions against mechanical strength and surface hardness of ceramic reinforced metal alloys and metal matrix composites. Applying the well-known alumina powders by dry pressing technology and some special pore-forming additives and sintering technology the authors have successfully developed a new, high porosity alumina matrix material for composites of advanced Al-Mg alloys. The developed new matrix material have higher than 30% porosity, with homogenous porous structure and pore sizes from few nano up to 2-3 mm depending on the alloys containments. Thanks to the used materials and the sintering conditions the authors could decrease the wetting angles less than 90° between the high porosity alumina matrix and the Al-Mg alloys.

Applied analytical methods in this research were laser granulometry, scanning electron microscopy, and X-ray diffraction. Digital image analysis was applied to microscopy results, to enhance the results of transformation.

Keywords: porous ceramics, alumina, wetting angle, matrix materials, composites

1. Introduction

Metal matrix composites (MMC) have been developed in recent years, among which aluminum matrix composites have found various applications in the industry. This is due to its low density, high toughness and corrosion resistance. [1,2]

It has been well established that the addition of ceramic particles to aluminum improves strength, wear resistance, and corrosion resistance. [3,4] Major applications include aerospace, military and car industries.

The need for composite materials has become a necessity for modern technology, due to the improved physical and mechanical properties, for example high strength [4], stabilization for chemicals, lighter than metal etc. [5,6,7,8]. However, ceramics are more brittle [9,10,11], than metals. So life time prediction is difficult. Because of this characteristic, the uses of ceramics are limited.

Recently, some composite materials of ceramics are developed such as “Particle reinforced composite material”, “Short fiber reinforced composite material” and “Continuous fiber reinforced composite material” [6,7]. As for these composite materials, ceramics aren’t main materials.

In this research, the composite materials are made by regarding porous ceramics as main materials. The porous ceramic is alumina. The porous ceramic samples were made by mixing various grain size and amount of additive materials with alumina. After making the porous ceramics, later, composite
materials are made by impregnating molten metal into the pores of ceramics (figure 2.) [12]. When a molten metal and a material have a contact angle less than 90 degrees by surface tension, the impregnation occurs spontaneously. [13,14,15]

However, when a molten metal and material have a contact angle bigger than 90 degrees by surface tension, impregnation doesn’t occur spontaneously. Here porous alumina and molten metal has contact angle less than 90 degrees so only the chemical reactions are used to impregnate molten metal into the alumina. The method is the “Primex method” [16,17]. It is used nitrogen as a chemical reaction. [18,19] But in this research we concentrate only on the making of matrix material of the composite, what is in this case porous alumina ceramic. Some researchers use the porous alumina ceramics to synthesing Al$_2$O$_3$-Al, Al$_2$O$_3$-Ti, Al$_2$O$_3$-Fe cermets or making Al$_2$O$_3$-Al powder composites. [20,21,22,23]

The main aim of this research is to make ceramic body from Al$_2$O$_3$ powder, as matrix material for the composite and this ceramic body reach at least 30 % porosity with adding organic pore forming materials to the Al$_2$O$_3$ powder. It is very important, if the molten metal is infiltrating in the ceramic across the pores, and so can impregnate it [24]. (figure 1.)

![Image](Figure 1. Producing of composite material)

At last, it is also very important, that the ceramic bodies reach the highest breaking strength near the high porosity.

2. Experimental
First of all, the properties of the used Al$_2$O$_3$ powder was determined. The main material is the Martinswerk KMS-94 type Al$_2$O$_3$ powder, which compound by X-Ray diffraction: Al$_2$O$_3$ : ~ 94%, CaO : ~ 0.3 %, SiO$_2$ : ~ 4 %, MgO : ~ 1 % . The average grain diameter was measured of the powder by laser granulometry, what was 104.62 μm and the counted specific surface was 0.106 m$^2$/g. (figure.2,3)

![Image](Figure 2. Grain size distribution of the used alumina powder)
![Image](Figure 3. Picture /SEM/ of the used Al$_2$O$_3$ powder)

After that, mixtures was made with the Al$_2$O$_3$ powder and various size and amount of organic pore forming additive materials. The compounds of the mixtures was determined to find the optimal level
of parameters to producing the matrix, which have high porosity and breaking strength (full factorial design of experiment):

| RunOrder | Pore forming size [um] | Pore forming amount [%] | Pressing force [N] |
|----------|------------------------|-------------------------|-------------------|
| 1        | 100                    | 1                       | 4000              |
| 2        | 300                    | 5                       | 4000              |
| 3        | 100                    | 5                       | 4000              |
| 4        | 300                    | 1                       | 4000              |
| 5        | 100                    | 5                       | 3000              |
| 6        | 300                    | 5                       | 3000              |
| 7        | 300                    | 1                       | 3000              |
| 8        | 100                    | 1                       | 3000              |

To each mixture was added some water and plastificator material. After that the mixtures stayed in bags for 72 hours. After making the mixtures, samples were formed by dry uniaxially pressing. Each pieces had 15 mm diameter and the pressing force was 3000 N and 4000 N by each forming, what means 16.98 MPa and 22.64 MPa pressing pressures. After the forming of the samples, they were sintered in the laboratory furnace where was $1350^\circ C$ the maximum temperature.

3. Results and discussion

Two properties of the samples were measured. The first property was the porosity of the sintered ceramic bodies. The determining of porosity values were by the following equation (1):

$$P_b = \frac{W_3 - W_1}{W_3 - W_2} \times 100 \quad [%]$$  \hspace{1cm} (1)

where are the weight of dried body ($W_1$), the weight of the wet body ($W_3$) and the weight of the body under the water ($W_2$).

After measuring porosity the breaking strength of the samples were measured with the mechanical pressing machine, but in this case was not registered the pressing press, but the breaking strengths. By the measuring the breaking strength were reached very impressive values near the high porosity.

The impact of the different factors for the porosity and the breaking strength and the optimal levels (vertical line) can be seen on the figure 4.

The main statements for the optimal levels of factors are the following:

- Pore forming additive size – impact on the porosity and breaking strength as well → middle level (~200 um grain size)
- Pore forming additive amount – major impact on the porosity → (~4 w/w%)
- Pressure – major impact on the breaking strength → high level (4000 N)

Applying the optimal levels of factors an alumina matrix material was prepared with 35% porosity and 14 MPa breaking strength.
Figure 4. The impact of the different factors for the porosity and the breaking strength and the optimal levels (vertical line)

At last, the broken samples were investigated with Scanning Electron Microscope, to compare our samples with the samples from the references, from other researches. On the figure 5 is the SEM picture of the broken Al$_2$O$_3$ ceramic from the reference, and on the figure 6 is the SEM picture of our, broken Al$_2$O$_3$ ceramic.

Figure 5. Microstructure /SEM/ of the samples sintered 1500 °C /5K/
From: Yong-sheng Han, Jian-bao Li, Ke Tang, The effect of sintering temperatures on alumina foam strength, Ceramics International 28 (2002) 755-759 [6]

Figure 6. Microstructure /SEM/ of our ceramic samples /5K/

It can be seen, that in our samples are more little grains and higher porosity than the sample from the reference. It is because our samples were sintered on lower temperature.
Finally, the Al$_2$O$_3$-Al composite was made by the above mentioned “Primex” method. The infiltration was gone off using nitrogen atmosphere and 1100 °C heating temperature by the infiltration. Thanks to these conditions, the molten metal and the ceramic material had a contact angle less than 90 degrees by surface tension and so the impregnation could occur spontaneously.

The chemical compound of the composite was investigated by X-ray diffraction. The compound of the composite was the following: Al: ~5%, Al$_2$O$_3$: ~80 %, and 15 % other components.

The microstructure of the composite material was investigated by Scanning Electron Microscopy. On the photographs can be seen, there is the Al metal /dark area/ around the Al$_2$O$_3$ /bright area/ particles (figure 7).

The hardness of the composite was determined applying a Vickers indenter and calculated as

\[ H = \frac{P}{2d^2} \]  

(2)

where \( d \) is the half-diagonal indentation impression and \( P \) is the indentation load (100 N for 15 s). The results of hardness measurements are very impressive, 1200 HV10 on the average. This is an expressly high hardness, if we compare to other references, where the authors have reached only 800-900 HV.

\[ \text{Figure 7. Microstructure /SEM/ of the Al}_2\text{O}_3 \text{ composite material, the bright area is Al}_2\text{O}_3 \text{ and the dark area is Al} \]

4. Conclusions

It could be seen, there were made high porosity Al$_2$O$_3$ ceramic bodies with more than 30% real porosity and 14 MPa breaking strength by adding organic pore forming additive materials.

The method of design of experiment can be successfully applied to get the optimal levels of factors for preparing a high porosity ceramic matrix material.

By preparing the matrix, the most significant impact on the porosity and breaking strength has the pore forming size (due the volume of pores), and the pressure has the less impact on the pores and breaking strength.

Our prepared samples are more little grains and higher porosity than the sample from the reference. It is because our samples were sintered on lower temperature and in nitrogen atmosphere.

It can be established, that the porosity is inversely proportional to the breaking strength.

By preparing the composite, the porous alumina and molten metal has contact angle less than 90 degrees, so the “Primex” method (spontaneous infiltrating) could occur.

The Al$_2$O$_3$-Al composite material was produced successfully, with extreme high hardness and relatively homogeneous microstructure.

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