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An Investigation of the Chemical Strength of Directionally Solidified MgAl₂O₄-Y₃Al₅O₁₂ Eutectic Ceramic Materials at Gas Turbine Conditions

Yönlü Katılaştırılmış MgAl₂O₄-Y₃Al₅O₁₂ Ötektik Seramik Malzemelerin Gaz Türbin Şartlarındaki Kimyasal Dayanımının İncelenmesi

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Abstract

Nickel based alloys are mostly use in aircraft components such as nozzle, blade and heat shield in gas turbine engines. But the resisting temperatures of these alloys are limiting their applications, and cause environmental pollution with increasing energy costs. However directionally solidified eutectic oxide ceramics grown following melting, may eliminate these problems. In this study, the comparison of the production and hot corrosion resistance of eutectic structure MgAl₂O₄-Y₃Al₅O₁₂ (MAS-YAG) produced by the directional solidification method and sintering magnesium aluminate spinel (MAS)-yttrium aluminium garnet (YAG) composite were compared for the same chemical composition. For this reason, volume, weight and microstructure (SEM) changes of the sinter and eutectic materials were observed. The flexural strength behaviour of sintered and eutectic MAS-YAG materials were investigated at room temperature and corrosive gas turbine condition at 1600°C. Corrosion results of directionally solidified MAS-YAG structure were better than conventional sinter ceramics for the same composition.

Keywords: Corrosion, Directional solidification, Eutectic oxide ceramics, Single crystal, Zone melting

Öz

Özellikle havacılık uygulamalarında kullanılan gaz türbin motorlarındaki nozul, bıçak ve ısı kalkanı gibi parçalarda Nikel esaslı alaşımlar kullanılmaktadır. Fakat bu alaşımların dayanım sıcaklıkları uygulamaları sınırlandırmakta, enerji maliyetini artırarak çevre kirliliğine neden olmaktadır. Ergitilerek büyütülen yönlü katılaştırılmış ötektik oksit seramikler ise bu olumsuzlukları bertaraf etmektedir. Bu çalışmada da yönlü katılaştırılmış MgAl₂O₄-Y₃Al₅O₁₂ (MAS-YAG) seramiklerinin üretimi ve sıcak korozyon dirençlerinin, sinterlenerek üretilmiş magnezyum alüminat spinel (MAS)-itriyum alüminyum garnet (YAG) kompoziti ile karşılaştırılması ele alınmıştır. Bu amaçla sinter ve ötektik malzemelerin hacim, ağırlık ve mikroyapı (SEM) değişimleri gözlenmiştir. Oda sıcaklığı ve korozif 1600°C'lik gaz türbin şartlarında sinterlenmiş ve ötektik MAS-YAG malzemelerin eğilme mukavemeti davranışları incelenmiştir. Yönlü katılaştırılmış MAS-YAG yapının korozyon davranışları aynı kompozisyondaki konvansiyonel sinter seramikten daha iyidir.

Anahtar Kelimeler: Korozyon, Yönlü katılaştırma, Ötektik oksit seramikler, Tek kristal, Ergiyik zon

1. Introduction

Ceramic and ceramic based composite materials have an important place in the use of structural materials in terms of temperature, abrasion resistance and oxidation resistance. However, the strength of sintered polycrystalline ceramic materials shows a sudden decrease with temperature increase (Ohnaka et al. 2002). Atmospheric conditions in alumina at high temperatures (water vapour) are known to cause growth of grain boundaries and deformation of material.

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On the other hand, it has been found that the directionally solidified YAG $(Y_3Al_5O_{12})$ material has very good corrosion resistance under the same conditions (Bahlawane and Watanebe 2000). Waku et al. have shown that Al_2O_3 -YAG materials can be grown in a single crystal by directional solidification by the Bridgman method (Waku et al. 1998). Because these materials do not contain grain boundaries and colonies, they exhibit superior thermal resistance at very high temperatures. Especially today, it is necessary to work at higher temperatures in order to increase the combustion efficiency of gas turbine engines. In this case, Ni-based alloys which are resistant to high temperatures used in the

nozzles and combustion chambers are already inadequate in gas turbine engines. Because the melting temperature of these materials is 1400°C and they are subject to sudden decomposition at 1000°C. For this reason, directionally solidified eutectic ceramic materials are being developed for use in combustion chambers to overcome the temperature limitations of metals. It is known that the US air force has decided to study these materials for use in turbine generators in the future (Courtright et al. 1992, Waku 1998). Nitride and carbide based advanced ceramics may not present sufficient strength in oxidizing environments at high temperatures. Therefore, directional solidification method concentrates on oxide eutectic ceramic materials. These materials exhibit ductile behaviour at high temperatures in gas turbine and power generator applications, but also maintain the strength at elevated temperature. In this study, high temperature chemical resistance of polycrystalline structures with the same composition as solidified MgAl₂O₄-Y₃Al₅O₁₂ was evaluated.

2. Materials and Method

2.1. Sample Preparation

In the study, the weight percentage of the eutectic composition of Al₂O₂ (99.99%) - MgO (99.99%) - Y₂O₂ (99.999%) powders is 55-25% of Al₂O₂-MgO by weight %. The powders of desired composition are ground using a vibrating micro mill (Retsch PM-100, Tubitak-MAM). Particle size and distribution were controlled by particle size analysis. (Mastersizer, Tubitak MAM KE). The powders were milled in a dry jet mill (Macchine-Macina-Smalto, COMU) after being sintered at 1000°C for 1 hour (atmosphere controlled furnace, ITU-KMF) for granulation. The prepared sample was formed with a cold press (ITU-KMF) at a compression rate of 320 MPa and was then sintered at 1300°C for 2 hours in the same furnace. After this step, the sample production was divided into two steps, the polycrystalline samples were discarded after the above-mentioned sintering process at 1300°C, and the MAS-YAG powders were synthesized using the float zone furnace shown in Figure 1 (JUTEM) at 20 mm/h growth rate, 10 mm diameter and 150 mm length. Author Abali (2014) identified the phase composition of the material as $MgAl_2O_4-Y_3Al_5O_{12}$ in his previous work. In this study, the chemical behavior of single crystal material was investigated in the same composition. The author focuses on the comparison of corrosion resistance of the material with the polycrystalline form.

2.2. Characterization

For high temperature corrosion resistance of polycrystalline and directionally solidified MgAl₂O₄-Y₃Al₅O₁₂ materials, the samples were cut into $3 \times 4 \times 40$ mm size and converted into 4 µm layer by ultrasound surface polishing with pure ethanol. Corrosion tests Al₂O₃ (99.8% purity) tube furnace was performed at 1600°C with a heating rate of 100°C/h for 72 hours and the temperature was controlled by a thermocouple (Pt% 13Rh-Pt). The tube and crucible were passed through with wet oxygen gas for 18 hours at 1600°C prior to the corrosion test. After corrosion tests, weight and volume changes of polycrystalline and directionally solidified MgAl₂O₄-Y₃Al₅O₁₂ crystals were evaluated. An electron microscope (JEOL JSM-6335F) was used for micro-image analysis of the fractured surface. Flexure strengths of sintered MAS-YAG and directionally solidified MAS-YAG eutectic single crystal ceramics were measured at room temperature and at gas turbine conditions (1600°C oxidizing atmosphere, 30 hours) (INSTRON 8801).

3. Results and Discussion

3.1. Particle Size Distribution

Figure 2 shows the results of grain size distribution analysis of micro milled pulverized powders. One of the most important criteria in the crystal growth process is that the average grain size should be in micron size and as homogeneous as possible. As can be seen from this figure, the average grain size of the powders is 18μ m. A narrow range of particle size distribution is a positive result for a uniform structure.

3.2. Chemical Strength

The mass change ratios are seen for both polycrystalline $MgAl_2O_4$ - $Y_3Al_5O_{12}$ and directionally solidified single crystalline $MgAl_2O_4$ - $Y_3Al_5O_{12}$ ceramics under the corrosive atmosphere (O_2 - H_2O) as given in Figure 3.

The mass change is very low when the single crystal is $MgAl_2O_4$ - $Y_3Al_5O_{12}$ whereas polycrystalline $MgAl_2O_4$ - $Y_3Al_5O_{12}$ has lost weight apparently. The deformation process is accelerated because the sintered material is deprived of interface. The hydroxide environment might have caused some fragmentation on surface of polycrystalline sample at high temperature, especially after 60 hours, resulting in some weight loss. That is, the sintered polycrystalline structure begins to deform. At the end of the corrosion test, the volume change rates of sintered and directionally solidified samples over time are shown in Figure 4.

The volume changes of the directionally solidified single crystal eutectic oxide composite ceramic $MgAl_2O_4/Y_3Al_5O_{12}$ is negligible. In the sintered polycrystalline form of the same composition, the corrosion test resulted in a visible loss of volume corresponding to loss of weight.

3.3. Microstructure

In this case, as can be seen in Figure 5 the pores in the sintered material gradually decreased, leading to a reduced



Figure 1. Zone melting furnace.



Figure 2. Particle size distribution of powdered sample.

volume due to shirinkage. This behaviour, which may have tragic consequences in the operating conditions of gas turbine engines, is a condition to be considered.

3.4. Mechanical Properties

Figure 6 shows the flexure strength behaviour of both materials depending on the temperature after corrosion test. The author (Abalı, 2014) in his previous work, showed the flexure strength of directionally solidified MAS-YAG ceramics without corrosion test.

The temperature effect is inherently an environmental factor and, together with the corrosive conditions, creates



Figure 3. Time dependent weight change rates of sinter and and directionally solidified eutectic ceramic.



Figure 4. Time dependent volume change rates of sinter and directionally solidified eutectic ceramic.



Figure 5. Microstructure analysis of polycrystal MgAl₂O₄- $Y_3Al_5O_{12}$ sample, **A)** before corrosion test **B)** after corrosion test.



Figure 6. Temperature dependence flexure strength of both materials under corrosive condition (red: sinter MAS-YAG – blue: DS MAS-YAG).

a situation that pushes the limits of resistance even further. It is known that both materials have higher flexure strength values without oxidizing atmosphere. As shown in the figure, the mechanical behaviour of the sintered and directionally solidified MAS-YAG structures, which have the same chemical structure at room temperatures up to 1600°C, are also different depending on the temperature subjected to the corrosive environment for the same time. Directionally solidified MAS-YAG maintains its strength at room temperature up to 1600 °C. The conventional sintered MAS-YAG structure is able to maintain a similar strength (400 MPa) up to 1000°C.

4. Conclusions

Directionally solidified MgAl₂O₄- $Y_3Al_5O_{12}$ ceramics have more strength to chemical conditions at 1600°C than the those produced by sintering. Today's Ni-based alloys cannot be used in gas turbine engines at high temperatures due to creep effect and corrosion. For nozzles and blades used in the hot zones of future shuttles, airplanes, jet engines and transatlantic engines, the materials to replace Ni-based alloys will most possibly be directionally solidified oxide eutectic ceramic composites.

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