COMPARISION OF AL_2O_3 + SIO_2 AND AL_2O_3 COATING AS THERMAL BARRIER IN AISI 4140 ALLOY

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Abstract:- Nozzle is one crucial part of rocket. In this research, the nozzle was made by AISI 4140 coated by thermal barrier coating (TBC). The TBC material is Al_2O_3–SiO_2 composite, with Al_2O_3 – 50% SiO_2 -50%. The aim of this research is to observe the composition which will produce optimum adhesive strength and the most widely formed mullite phase. Mullite is an expected phase formed from composite and it has thermal resistance even during 1300˚C. Coating process was conducted by flame spray with variation of plies. The effect ratio, plies, microstructure, phase, and adhesive strength of TBC were investigated using Scanning Electron Microscope (SEM), X-ray diffraction (XRD), and adhesion testing.

Key Words**:** ***Nozzle, AISI 4340, Al_2O_3 – SiO_2, Flame Spray***.

1. INTRODUCTION

Nozzle contributes 50% from total weight of rocket structure. Nozzle is a part of rocket in which hot gas produced in the combustion chamber is permitted to escape throughout the combustion chamber. Nozzle converts potential energy of hot gas flow into thrust. High pressure and thermal of combustion gas coming out the limit of material used. This research used AISI 4340 steel for material base of nozzle and this steel meet material required for nozzle application. The AISI 4140 is Nickel – Chrome – Molybdenum alloy with good hardenability, high strength and toughness [1]. However AISI 4140 has lower melting point as compared to required temperature (*ca.*1300-1700^{0}C) [2]. In addition, coating of AISI 4140 by Thermal Barrier Coating (TBC) can be the solution to overcome the lack of thermal resistance of material. All the coatings were plasma sprays onto mild steel substrates with thick Nickel Aluminide (NiAl) bond coat. TBC is one solution for high temperature resistance application, it consists on ceramic on top coat and bond coat. Ceramic is stable material at high temperature, some of ceramic material for TBC applications were Al_2O_3 (Alumina) and SiO_2 (Silica) [3]. Mullite is one of phase combination between SiO_2 and Al_2O_3 able to resist high temperature (*ca.* 1400-1600^{o}C). This paper reviews the analysis of TBC of Al_2O_3 - SiO_2 coated on AISI 4140 by flame spray.

Methodology

Al_2O_3 - SiO_2 coatings were deposited on substrate using a flame spray technique. Briefly 100 x 30 x 6 mm AISI 4140 was positioned in the throat of nozzle. Before coating, grit blasting conducted on substrate to enhance the roughness of surface [4]. The material used in grit blasting is aluminium brown oxide powder with composition coarser < 20%, basic > 45%, mixed > 70% and finer < 3%. Chemical composition of grit blasting powder were 60.39%, 9.45%, 4.97%, and 11.18% for SiO_2,Fe_2O_3, TiO_2, and Al_2O_3 respectively with 5.5 bar in pressure. The roughness of surface after grit blasting is approximately 40.8 – 56.2 µm. Bond coat (NiCH) coated by flame spray on the surface to inhibit oxidation in intermediate substrate at high temperature. The bond coat is arguably the most crucial component of TBC system. Its chemistry and microstructure influence durability through the structure and morphology of TBC. During coating process, aluminium will oxidize to form passive layer (aluminium oxide) and inhibit further oxidation. Furthermore, bond coat also increase adhesivity between substrate and topcoat [5]. The Al_2O_3-SiO_2 powder was used as coating material. Particle size of Al_2O_3 powders were 89.67 µm, while SiO_2 powders were 56.59 µm, both of them were in polygonal shape. They were mixed based on different ratio of SiO_2: 20, 30, and 40% respectively. Using ball milling 400PM with 350 rpm for 1 hour. Coating process conducted by Flame Spray for 2, 4, and 6 plies by assuming 1 plies for 20µm. Flame Spray process parameters were the gas flow rate (0.1 mm/h), rate powder (0.79/0.1 mm, kgm^2/jam), spray distance (130-180mm), air pressure (5000 psi), oxygen pressure (30kg/cm^2), pressure asetelin (30kg/cm^2) and flame temperature 1200^{o}C. The phase analysis, microstructure and adhesive strength of TBC after coating were measured by X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Pull-off test Fixed-Alignment Portable Tester Type II before and after heating process. Thermal resistance measured after heating process of TBC at temperature 1400^{0}C for 14 second.

Result and Discussion

A. Characterization of Al2O3 – SiO2 powder

Powder Al_2O_3 and SiO_2 were mixed in accordance with the composition using a mechanical process. The microstructure of mixing powder

Distribution of powder after mixing with mixing ratio 50% Al_2O_3 + 50% SiO_2

The particles tend to agglomerate with the increase of SiO_2 content due to the different particle size and surface roughness; particle size of SiO_2 is smaller than Al_2O_3 and the SiO_2 Surface area is more roughness than Al_2O_3. After flame spray, TBC from Al_2O_3-SiO_2 powder was observed by SEM as shown in TBC protect the underlying base metal by removing the lower melting point base material from the high heat flux/temperature regions. Refractory metal bond coatings are required to create a good bonding between the ceramic top coating and the base metal. Bonding qualities depend on homogeneity of microstructure of TBC powder. Several phenomena of unhomogen on top coat of TBC microstructure. TBC powder look like melted, semi melted and unmelted. This phenomenon was caused by flame temperature and insufficient time to melt all of coating powder during spraying.

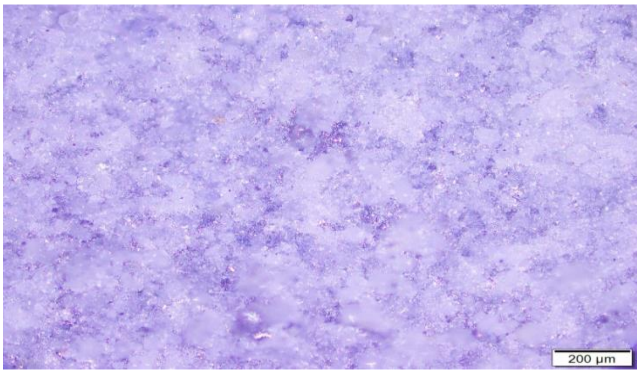

Fig 1. Microstructure of top coat Al_2O_3+SiO_2

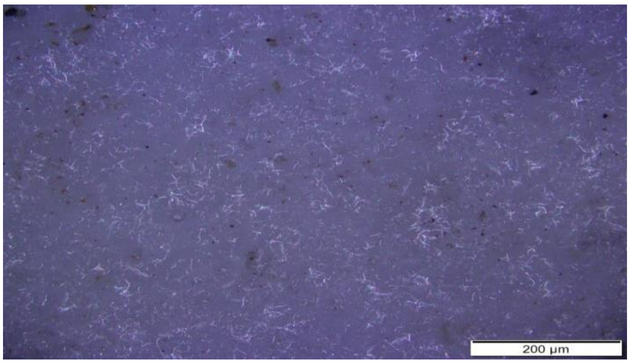

Fig 2. Microstructure of top coat Al_2O_3

Fig 1. Microstructure of top coat for the Al_2O_3+SiO_2 show the color image to the structure of the coating
With the image of the 200 µm

Fig 2 Microstructure of top coat for the Al_2O_3 show the color image to the structure of the coating with the image of the 200 µm

Average thickness of top coat for 6 plies is 407,5 µm However, after heat treatment process, the thickness of top coat was decrease and the amount of porosity was formed. The difference of coefficient thermal expansion between substrate, bond coat and top coat material initiate the porosity. Porosity existence results in reducing particle bonding and triggering oxidation

B. Thermal Barrier Coating characterization by XRD

After coating process of AISI 4140 by NiCH and Al_2O_3-SiO_2, phase characterization was conducted by X-ray diffraction (XRD). The same pattern for all different composition of Al_2O_3 -SiO_2. The identified phase were Al_2O_3, SiO_2, Al_2SiO_5 (Andalusite), Kyanite ($Al_2O_3 \cdot SiO_2$) and NiCH. Kyanite is one of Al_2O_3-SiO_2 polymorphs formed in the range 200-800°C, while mullite phase could not be identified due to the lack of uniform heating during flame spray process in which the temperature was 1200°C. Moreover, mullite formed at range temperature above 1550°C. After heat treatment process, the TBC phase was characterized as well and presented in. XRD pattern of TBC before heat treatment for 50% Al_2O_3 + 50% The XRD pattern becomes amorphous. Small peak of mullite formed at 2θ were in 26.27° and 40.88°, in accordance with the JCPDS card number 79-1455. This result is in agreement with the previous research conducted by Dilip Jain which states that the presence of Kyanite and alumina phase will be able to trigger mullite forming at temperatures below 1550°C.

C. Adhesive Strength Characterization

Adhesive strength defined as bonding strength between substrate and coating layer. Adhesive strength between TBC (Al_2O_3-SiO_2) on AISI 4140. Decreasing adhesive strength of TBC occurs due to increasing of SiO_2 content, and it was caused by the presence of porosity. Since the particle shapes of SiO_2 were in angular and roughness, it can trigger the porosity during spraying process. Based on adhesive test knowing that powder which more percentage of SiO_2

Has lower adhesive strength. That caused by the shape of SiO_2 powders are angular and aggregate particulate which have roughness surface and caused more porosity. Therefore, it has lower particulate bonding. The more SiO_2 powder, the less particulate bonding for SiO_2 and Al_2O_3 or NiCH. It was very important for measuring TBCs as their function is to provide a thermal gradient between the outside and the inside of the coating and hence depth discrimination is essential. The interface between TBC and bond coat is another critical element.

Summary

Thermal Barrier Coating prepared from Al_2O_3 – SiO_2 had been successfully coated on AISI 4140. The optimum adhesive strength was 16 MPa with mixing ratio 50% Al_2O_3 – 50% SiO_2. Mullite formed initially at mixing ratio 70% Al_2O_3 – 30% SiO_2, while Kyanite (Al_2O_3•SiO_2) and alusite (Al_2SiO_5) were another new phases formed in TBC of Al_2O_3-SiO_2 which is produced by flame spray

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