Fabrication of High Thermal Conductivity NARloy-Z-Diamond Composite Combustion Chamber Liner for Advanced Rocket Engines

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Extended Abstract

Introduction

This paper describes the process development for fabricating a high thermal conductivity NARloy-Z-Diamond composite (NARloy-Z-D) combustion chamber liner for application in advanced rocket engines. The fabrication process is challenging and this paper presents some details of these challenges and approaches used to address them. Prior research conducted at NASA-MSFC [Ref. 1] and Penn State (Ref. 2) had shown that NARloy-Z-40%D composite material has significantly higher thermal conductivity than the state of the art NARloy-Z alloy. Furthermore, NARloy-Z-40 %D is much lighter than NARloy-Z. These attributes help to improve the performance of the advanced rocket engines. Increased thermal conductivity will directly translate into increased turbopump power, increased chamber pressure for improved thrust and specific impulse.

Early work on NARloy-Z-D composites used the Field Assisted Sintering Technology (FAST, Ref. 1, 2) for fabricating discs. NARloy-Z-D composites containing 10, 20 and 40vol% of high thermal conductivity diamond powder were investigated. Thermal conductivity (TC) data are shown in Figure 1. TC increased with increasing diamond content and showed 50% improvement over pure copper at 40vol% diamond. This composition was selected for fabricating the combustion chamber liner using the FAST technique.

Combustion Chamber Liner Design

A subscale combustion chamber liner was designed to fit in an existing hot fire test assembly at Marshall Space Flight Center (MSFC) shown in Figure 2. It is basically a 2.75 in OD, 2.50 in ID and 8 in. long cylinder with built in cooling channels on the outside. It is over coated with electroformed nickel and welded to the test assembly. This paper focuses on the fabrication of the liner.

Fabrication Process

The FAST process was used to make the liner. It is a powder metallurgy process in which NARloy-Z powder is blended with diamond powder, poured into a mold and sintered under pressure to produce a full density part. Since the FAST process is limited to 1.5 inch tall cylinders, it is planned to make 6 rings (1.33 in. tall) and diffusion bond them to form the 8 inch tall liner. Cooling channels will be machined on the outer surface of the liner by a machining vendor.

Initially, mold, core and die were and fabricated from graphite. NARloy-Z and diamond powder were mixed and poured into the mold, placed in the FAST apparatus (Figure 3A) and consolidated to full density (Figure 3B). Two plates of NARloy-Z-D were diffusion bonded together (Figure 4A) using the same FAST parameters and tested for bond strength. In addition, a cylindrical ring (Figure 4B) was fabricated to demonstrate feasibility.

Fabrication Challenges

1. *Machining of NARloy-Z-D Composite Liner*

Diamond is the hardest substance known and NARloy-Z-D composite is not much different. It is too hard to machine by conventional means. Electrical discharge machining (EDM) and water jet cutting/grinding techniques seem to work satisfactorily. Both methods were used for cutting test specimens. Machining channels proved to be more challenging and required special tooling. Both EDM and water jet techniques were evaluated for grinding channels by different vendors. The results so far indicate that water jet grinding gives better results than EDM for machining channels.

2. *Net Shape Forming of NARloy-Z-D Liner*

Penn State made an evaluation of forming the liner net shape that included the cooling channels. This design proved to be much more difficult to process by FAST. The narrow channels made the graphite mold very weak and susceptible to breakage under processing loads. To alleviate this problem TZM (tungsten-molybdenum) alloy was used for mold and die. A simple hollow cylindrical shape (Figure 4B) was used for initial trials. The mold and die material worked reasonably well but experienced a galling problem when the mold and die surfaces rubbed against each other. Galling produced rough surfaces in the part and also damaged the molds. To solve this problem mold-die clearances were increased slightly and a dry film lubricant (graphite foil) was applied to eliminate galling. This procedure could not be used on grooved molds since the grooves were too narrow for this technique to work. On detailed examination it was concluded that net shape forming with integral channels is extremely difficult to achieve without significant development work. Therefore a decision was made to go with a simpler, near net shape liner, i.e., a straight cylinder of appropriate thickness and height by diffusion bonding cylindrical rings.

3. *Segregation of Diamond Powder in NARloy-Z-D composite*

Penn State uses an acoustic mixer to mix NARloy-Z and diamond powders. This homogeneous mixture is then poured into the mold and consolidated. Early results showed that this mixture tended to segregate badly during consolidation (Figure 4A). Segregated areas showed mostly diamond particles and very little NARloy-Z. This segregation lowered both mechanical and thermal properties significantly and must be avoided. To alleviate the problem acoustic mixing was combined with a rotational mixing process (called 'turbula') to improve homogeneity but the mixture still segregated during pouring into the mold. Diamond powder is so slick that segregation is very easy. Tendency for segregation is so great that even blending with an organic binder did not help. The binder did not adhere to the diamond surface. Therefore it was concluded that it is necessary to pre-coat diamond powder with a thermally conductive coating to make it mix better with NARloy-Z powder. At the same time the coating should form a thermally conducting metal carbide layer on diamond particles. Commercially available titanium coated diamond powder was mixed with NARloy-Z powder and consolidated. The resulting microstructure was much more homogenous than using uncoated diamond powder.

4. *Nondestructive Examination of NARloy-Z-D Composite*

The segregation issues described above first surfaced when cutting the test specimens. Segregated areas were observed on the cut specimens that was visible to the naked eye. Examination under microscope showed areas of heavy segregation (Figure 5). It became apparent that a reliable nondestructive technique was needed to assure the homogeneity of the liner that must be free from internal defects. Computed Tomography (CT) technique was used on a solid block of NARloy-Z-D and a liner ring, both made by FAST process. The CT technique worked satisfactorily in revealing segregated volumes (Figure 4 A, B). Now it serves as a standard technique for verifying soundness of NARloy-Z-D chamber liner.

Results

1. *Mechanical properties*

<u>Tensile properties:</u> Preliminary tensile properties were obtained for NARloy-Z-30vol%D and NARloy-Z-40vol% plates. The sample had segregated areas that gave poor results. Good areas gave much better properties. Tensile strength was 18-19 ksi for NARloy-Z-30%D and 18-24 ksi for NARloy-Z-40%D. These numbers are acceptable for the chamber liner application. Ductility was low at 1-2% elongation, which is low but not unusual for a composite. Improved mixing should give us better properties.

Limited elevated temperature tests were conducted at 935^{o}F. The tensile strength was 11 ksi, which is acceptable. Strength seems to be controlled by the matrix, not by diamonds.

<u>Diffusion bond strength:</u> NARloy-Z-D was diffusion boned with and without an interlayer of NARloy-Z-D powder. Bond strength was much better for joints made with NARloy-Z-D powder

inter layer, ~ 11 ksi, which is high enough to proceed with testing. Process optimization will continue to further improve the bond strength.

2. *Microstructure Analysis*

Microstructure was examined in both optical and scanning electron microscopes (SEM). Apart from segregation of diamond in some areas as discussed earlier, microstructure was normal elsewhere and showed fairly uniform distribution of diamonds (Figure 6).

3. *Thermal Conductivity*

Thermal conductivity measurements were made by Enrique Jackson/EM10 using an optical flash method (Ref. 3). Thermal conductivity (TC) results were much lower than expected. The low numbers were attributed to segregation of diamonds in the matrix. Improving the diamond distribution in the NARloy-Z matrix should help to improve both thermal and mechanical properties. Coated diamonds were used to alleviate the segregation problem as described in the next sections.

a. Titanium coated diamonds

Titanium coated diamonds were supplied by Sandvik Hyperion (formerly Diamond Innovations) of Worthington, Ohio. Coatings were 6-7 microns thick. The coated powder was mixed with NARloy-Z powder. The mixture was much more homogeneous than straight diamonds. This mixture was sintered using FAST. The resulting microstructure was very good - shown in Figure 6B. Thermal conductivity was measured and found to be significantly lower, however, suggesting that titanium acted as an insulator, probably due to the surface oxide layer that did not dissolve in the matrix and prevented the diamond from contributing to the thermal conductivity. Therefore a better approach was needed as described below.

b. MoC Coated Diamonds

Global Technology Enterprises have developed a technique of coating diamonds with refractory metal carbides by using a chemical vapor reaction process. They provided us with refractory metal carbide coated diamonds for this investigation. Diamond powder was initially coated with MoC and then over coated with copper. This powder was blended with NARloy-Z powder and sintered using FAST. The resulting composite was examined for segregation using CT, and found to be homogeneous. This material is being evaluated for thermal and mechanical properties.

Planned work

It is planned to complete the work with MoC coated diamonds in the next few months and produce the 8 in. long subscale chamber liner. Cooling channels will be machined and the liner will be integrated with test fixture. Results will be reported and discussed in the final version of this paper.

References

1. B. N. Bhat, et al, "Copper-Multiwall Carbon Nanotubes and Copper-Diamond Composites for Advanced Rocket Engines", JANNAF Liquid Propulsion Meeting, April 2013.
2. A. Rape, X. Liu, A. Kulkarni and J. Singh, "Alloy development for highly conductive thermal management materials using copper-diamond composites fabricated by field assisted sintering technology", Journal of Materials Science, February 2013, vol. 48, Issue 3, pp1262-1267.
3. E. Jackson, "Through-thickness Thermal Diffusivity and Through-thickness Thermal Conductivity Characterization of NARloy-Z-Diamond Specimens", NASA-MSFC, Materials and Processes Laboratory, Material Test Branch (EM10) Memorandum dated August 26, 2014.

Figures: Given below

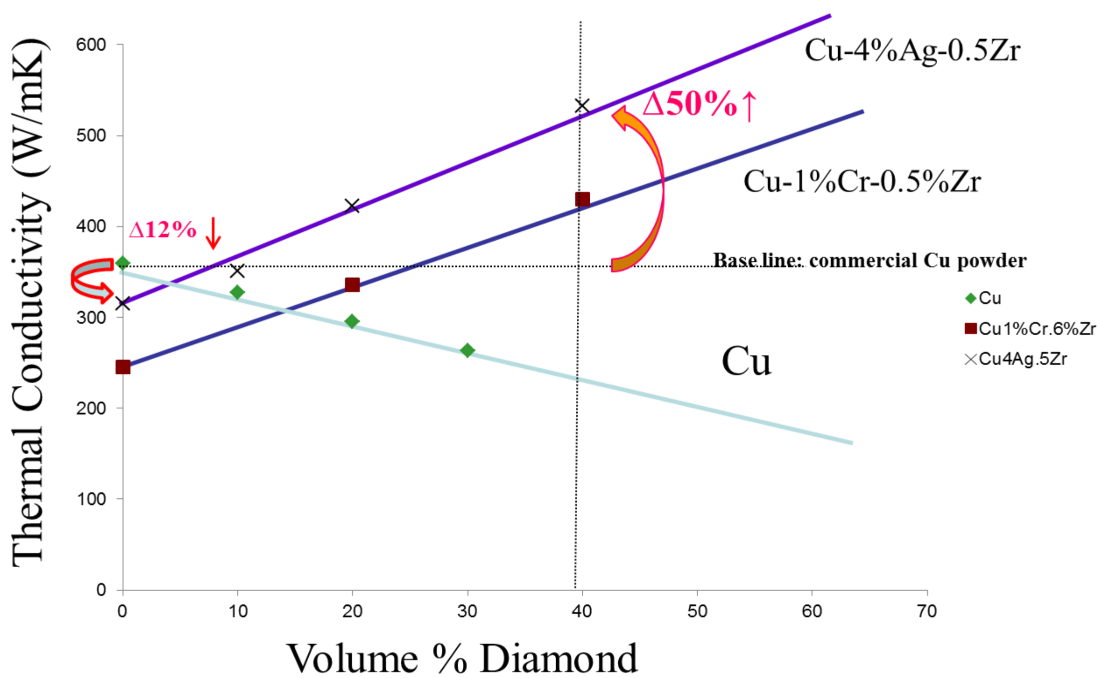


Figure 1: Thermal conductivity enhancement in Cu-Ag-Zr-D (NARloy-Z-D) composite [Ref. 2]

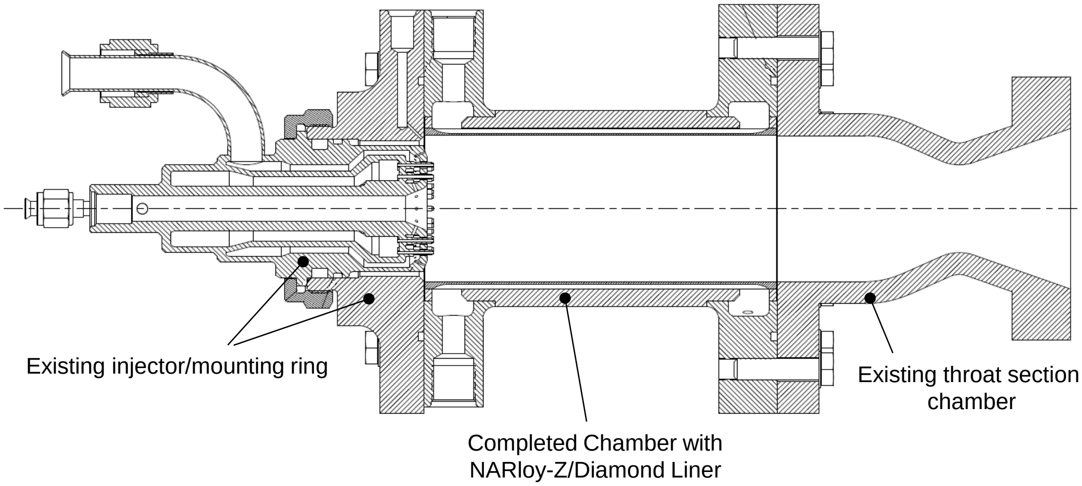
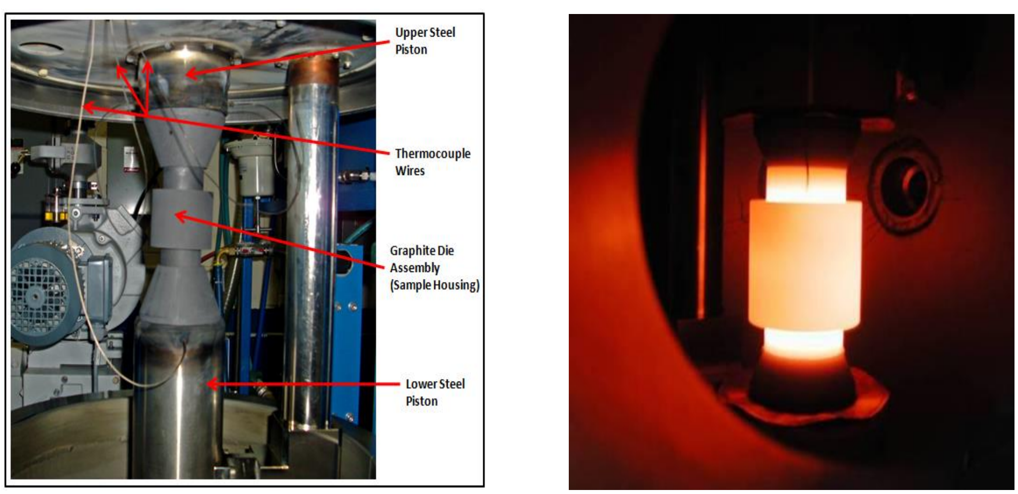
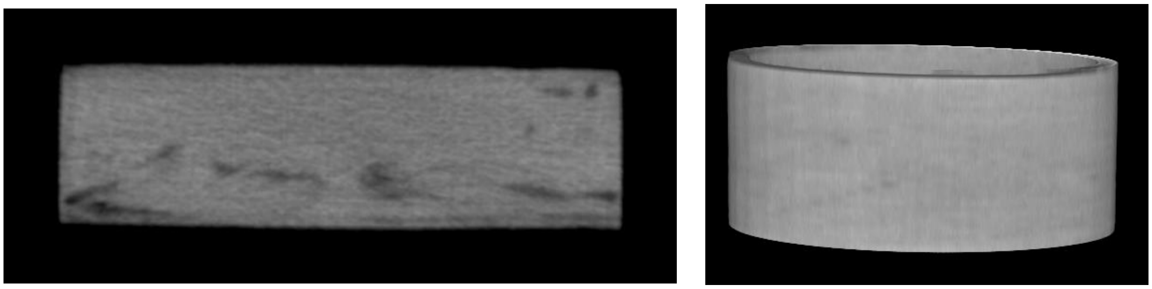


Figure 2: Hot Fire Test assembly schematic



A B

Figure 3: FAST system at Penn State Applied Research Laboratory (A); sintering at high temperature (B)



A B

Figure 4: Computed Tomography (CT) images of NARloy-Z-40%D composite plate (A) and ring (B) showing segregation of diamonds. Diamond segregation is indicated by dark areas. Segregation is more pronounced in A compared to B.

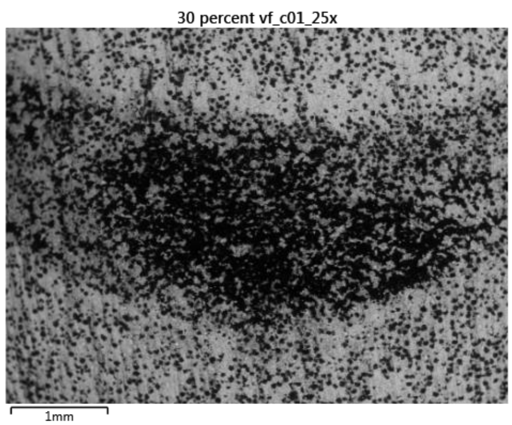
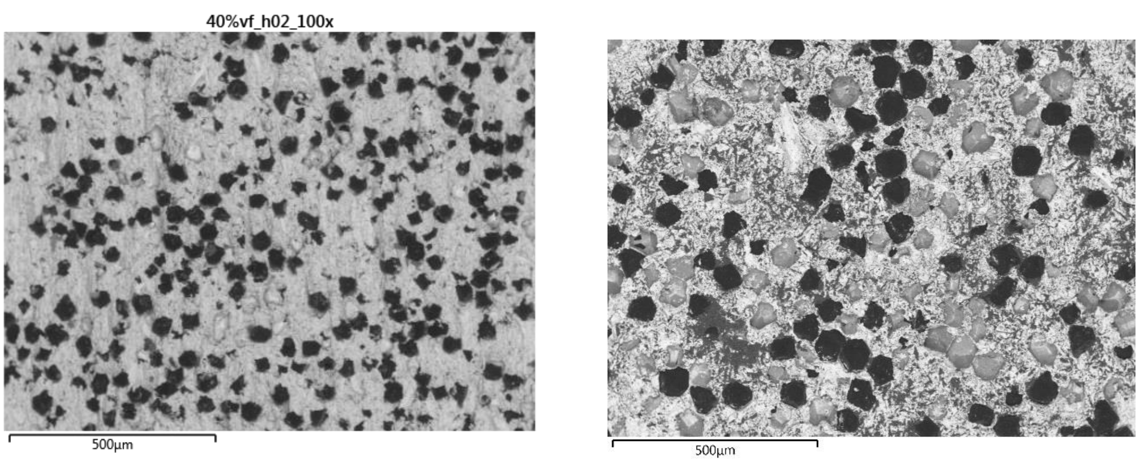


Figure 5: SEM Micrograph of Diamond segregation (dark area) observed in NARloy-Z-D Composite



A B

Figure 6: SEM micrograph of NARloy-Z-40% Diamond – without coating (A) and 40%Ti-coated Diamond composite (B). Titanium coated diamonds appear gray. Coating has come off in the dark looking diamonds during cutting