

# Development and Evaluation of Mechanical properties of subzero treated Al 6061-ZrO<sub>2</sub> MMC

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**Abstract**— *In the present study, Al6061-ZrO<sub>2</sub> metal matrix composites were developed by making use of the stir casting technique. The dispersed particle size was from 33 microns to 44 microns. The particles were added to the base material from 2 wt% to 8 in an increment of 2 wt% each. ZrO<sub>2</sub> particles were introduced to the matrix melt, the stirrer was used to create a vortex, which enhances the particle distribution into the matrix. The melt temperature was kept between 800 to 850°C. These composites were subjected to subzero treatment (Cryotreatment) i.e., the composite specimens were subjected to a freezing temperature of -196°C in liquid nitrogen gas (N<sub>2</sub>). The composites developed have a homogeneous distribution of reinforcement in the matrix alloy, with significant grain refinement and residual porosity, according to microstructural investigations. When compared to the matrix, mechanical characteristics show that the addition of ZrO<sub>2</sub> particles improved strength and hardness while reducing ductility slightly.*

**Index Terms**- Metal matrix composites, Al6061, ZrO<sub>2</sub>, Sub zero treatment and Stir casting technique.

## I. INTRODUCTION

In terms of material features like stiffness, specific strength, abrasion, and corrosion resistance, Metal matrix composites (MMCs) outperform pure metals and alloys allowing them to be used in cutting tools, aerospace, blades of the turbine, high-temperature components, and brake discs [1]. Metal matrix composites combine a mix of qualities of the metal matrix (aluminium, copper, or titanium) with high ductility and toughness, along with a hard ceramic reinforcing phase with high strength and modulus in the form of filaments, fibres, or particles. The mechanical performance like the strength, modulus, and creep life is improved while the wear is reduced [2]. The microstructures that form during the processing are largely responsible for the essential characteristics of MMCs. MMCs are often made in different ways including liquid state, solid-state, and in-situ processing solid-state processing like powder blending, consolidation, and diffusion bonding, which

is far costlier and more sophisticated than liquid-state processing [3,4]. Researchers have investigated different aspects of MMC manufacture, adjusted the structure and quality, and introduced better metal matrix Composites (MMCs), along with other things, in response to those issues.

Of the several MMC production methods discussed, stir casting or vortex casting is best for producing near-shaped final products. Vortex casting has so gained attention from the standpoint of commercial uses and academic investigations to form various MMC components with distinct mechanical and structural qualities. The stir casting method is considered to be a flexible, easy, and economical method for the production of MMCs [5, 6]. The likelihood of the second phase reacting with the melt, the likelihood for the second phase to settle down during casting, and the formation of reinforced phase accumulation are all disadvantages of this method. [7]. The increase of the properties of the metal-based composites need the proper diffusion of ceramic particles in the metal matrix composites and the attainment of strong physical attachments between the particles and matrix [8].

To decrease the clustering, interfacial reactions, gas cavities and proper distribution of reinforcement. The stirring speed, stirring time, ceramic particle feed rate, melting temperature, and mould temperature are all critical parameters to be considered during the stir casting process. [9, 10].

Aluminium metal matrix composites are significant MMCs due to their special properties like low density, a better balance of strength, ductility, formability, great corrosion resistance, and so on. These reinforcements all have one thing in common: they improve the aluminium matrix's mechanical characteristics and wear resistance. The ultra-low temperature processing of Al-ZrO<sub>2</sub> to improve

desirable metallurgical and mechanical properties is known as a cryogenic treatment. To achieve ultra-cold temperatures, computer controls, a well-insulated treatment chamber, and liquid nitrogen are used (LN<sub>2</sub>). This procedure is completely eco-friendly and helps to reduce trash. Depending on the weight and type of material being treated, the full procedure can take anywhere from 10 to 40 hours.

## II. LITERATURE REVIEW

The actual reason for the literature review is to attain an understanding of the research work that has been done in the domain area of the current study and to identify gaps in the literature related to Al6061 alloy/ZrO<sub>2</sub> particle composites. The literature survey has been conducted for over thirty years; the highlights of the literature survey are listed here. Because of its exceptional characteristics, AL6061 is the most commonly used aluminium alloy among numerous series. Al6061 is an alloy aluminium, magnesium, and silicon that is extremely corrosion resistant and has moderate strength [11]. With increased silicon carbide particles, Al6061 shows a rise in hardness and a decrease in ductility [12].

Stir casting allows zircon particles to be uniformly disseminated in Al- 4.5 wt% Cu alloy, and the abrasive wear resistance increases as the amount of particle and particle size decreases [13]. The properties of Al6063 reinforced with zircon sand and alumina were investigated. The results show that hybrid reinforced composites outperform Zircon and Alumina particle reinforced composites in terms of property improvement [14].

In a study comparing the hardness and tensile strength of zircon particles and TiB<sub>2</sub> Reinforced Al-AL6061 Alloy Matrix Composites to monolithic alloys, composites had better mechanical parameters and microstructure behaviour. Microstructures of zircon reinforced composites demonstrate uniform distribution particles and better bonding than TiB<sub>2</sub>, even though increasing the amount of reinforcement improves the conditions in TiB<sub>2</sub> reinforced composites [15]. Because a higher percentage of zircon dioxide in the composites causes particle agglomeration, stir casting is the better alternative. In stir casting, particles commonly clump together and can only be separated by strong swirling at high temperatures [16, 17].

Stir casting was used to incorporate zircon particles of various sizes and amounts into an Al-4.5 wt. per cent Cu alloy. The cellular structure of the composite matrix is determined by the size of the zircon particle and the amount of zircon in the composite. The abrasive wear resistance of composite improves as the amount or size of zircon particles is increased or decreased [18]. The addition of ZrO<sub>2</sub> particles to the Al 2124 alloy boosted strength and hardness while lowering toughness overall. As the proportion of ZrO<sub>2</sub> increased, the impact energy and percentage elongation dropped. Due to the growing ceramic phase of the matrix alloy, the hardness of MMCs increases approximately linearly with the volume proportion of Sic particles in the alloy matrix. Zircon dioxide (ZrO<sub>2</sub>), on the other hand, is a highly hard ceramic substance with a Mohr hardness of 7 to 8 [19].

## III. EXPERIMENTAL METHODOLOGY

In the electric resistance furnace, the matrix alloy Al6061 is heated to a temperature of 720°C. To induce slag development, scum powder is added to the molten metal, and the resulting slag is then removed from the melt. Degasification is accomplished by adding Hexachloroethane tablets to the melt while maintaining a temperature of 730°C. To form a vortex in the liquid metal, the stirrer is activated and set to rotate at 300 rpm. ZrO<sub>2</sub> reinforcement material was applied to molten metal after it had been preheated to 400°C. After 15 minutes of stirring, the molten metal is put into a 350°C pre-heated metal mould and allowed to solidify. Composites are designed for four distinct reinforcing weight percentages (ZrO<sub>2</sub>): 2, 4, 6, and 8 wt. %. The complicated capacity of the casting technique, which incorporates many processing factors such as stirrer speed, the temperature of processing, speed of pouring, the temperature of the mould, and feed rate reinforcement, determines the microstructure of ny material. The production of composites is regarded as the most complex and troublesome effort along these lines. As a result, Al6061-ZrO<sub>2</sub> MMCs are made using the stir casting method. Fabrication took place in a three-stage resistance sort 12 KW limit furnace. The furnace has a maximum temperature of 12000 °C and a temperature precision of 100 °C. It also has a partially integrated differential digital temperature controller and a seven-segmented light-emitting diode readout. On the inside, it contains an alumina melting pot that can be rotated

90 degrees on its orientation axis to allow the melt to be poured.



Fig 3.1 Electrical Resistance Furnace

The ZrO<sub>2</sub> particulate (44 microns) was preheated in a muffle furnace to a maximum temperature of 4000°C over for around 1 hour. The reinforcement must be preheated to lower the temperature slope and to enhance the wetting between the liquid metal and particle support. Al6061 composite has a melting point of between 550 and 62000 degrees Celsius. For ten minutes, a known number of Al6061 ingots were treated in a 10% NaOH solution at room temperature.

To remove the surface taints, pickling was utilized. The filth was cleaned by soaking the ingots in a solution of one part Nitric acid and one part water for a few seconds, followed by a methanol wash. The pickled ingots were dried and placed in the crucible to melt. The melt was superheated to 7200 degrees Celsius. Thermocouples were used to record the temperatures. Precautionary Measures and Confinements to be taken, the maximum temperature was maintained around. 7200C, above which it gets supersaturated and alumni fumes start emanating from the molten metal beyond 7500C. Due to density differences lot of care was taken in blending the reinforcement with the matrix with continuous stirring and then pouring the melt into the mould. Due to melt rejection at the mixing stage, composites with more than 10% reinforcement could not be made. This is now undergoing Cryogenic therapy.

To optimize its favourable metallurgical and mechanical characteristics, Al 6061-ZrO<sub>2</sub> is processed at an ultra-low temperature of roughly – 196°C. To achieve ultra-cold temperatures, computer controls, a

well-insulated treatment chamber, and liquid nitrogen are used (LN<sub>2</sub>). This procedure is completely eco-friendly and helps to reduce trash. The entire treatment can take anywhere from 10 to 40 hours, depending on the weight and type of material being treated.



Fig 3.2 Cryogenic Chamber

The technique affects the entire mass of the instrument or component being treated, making it stronger throughout. The hardness of the treated material remains unaffected, but its strength is increased.

#### IV. EXPERIMENTAL STUDIES

The purpose of this study is to determine the mechanical properties, microstructure, and XRD, of ZrO<sub>2</sub> reinforced Al 356 metal matrix composite. SEM inspection of tensile fractured specimens was performed to understand the type of failure.

##### A. Specimen Preparation for Microstructure Studies

Specimens for the microscopic study were developed as per ASTM E3 norms. The sample was initially grinded & polished and then etched. ZrO<sub>2</sub> grit paper (100-1200 grit size) was used to polish the specimens. The samples were held close by and rubbed easily against the ZrO<sub>2</sub> papers, practising adequate consideration to maintain a strategic distance from any profound scratches, because Al is very soft. MgO<sub>2</sub> glue was used for fine polishing, followed by diamond glue (1m thin) on the polishing equipment depicted in Fig 5.1. The billiard cloth was used to cover the platform. Hands and specimens were washed with water in between fine polishing with magnesium oxide paste to eliminate carryover of coarser grit from prior processes. The specimens were washed in alcohol and then dried outside in the sun.

**B. Etching**

200 grams of Chromic acid ( $Cr H_2O_4$ ) & 15 grams of Sodium Sulphate ( $Na_2SO_4$ ) & 1 litre of  $H_2O$  were utilized as an etchant.

**C. Optical Microscopy**

Optical micrographs were viewed with the help of Nikon Microscope LV150 Clemex Image Analyzer (reflection type), fixed to a camera Fig3.3



Fig 3.3 Optical Microscope

**D. Specimens prepared for Microstructure studies**

Specimens for microstructural investigations were cut from castings to diameter 10 mm and height 15 mm (Fig 5.3). They were polished using fine alumina powder as an abrasive and a succession of silicon carbide emery papers ranging in grade from 150 to 600. The specimens were microstructured using a computer-integrated metallurgical microscope. Micrographs were taken using a metallurgical microscope. The magnification ranged from 50 to 500 times.

**E. Specimen Preparation for Mechanical Characterization**

The prepared castings were machined into specimens with the required dimensions, with the center region of the castings being the most usual choice. All specimens were polished for a smooth surface finish with various grits of  $ZrO_2$  carbide sheets, except for those required for microscopic study, which required extra polishing. The following are the characteristics of specimens prepared for various tests:

SL No	Type of Test	Specimen Dimensions
1	Tensile	Diameter 12.5 mm and gauge length 65 mm
2	Compression	Diameter 20mm and length 20 mm

3	Hardness	Diameter 20mm and length 20 mm
4	SEM	5mm X 5mm X 1mm

**V. MECHANICAL PROPERTIES EVALUATION**

**A. Hardness test**

By measuring the depth of penetration of an indenter loaded on a material test piece, the Brinell scale quantifies the indentation hardness of materials. In materials science, it is one of the numerous hardness definitions. Johan August Brinell, a Swedish engineer, developed the first widely used and standardized hardness test in engineering and metallurgy. The size of the indentation and the risk of damage to the test item limit its utility. The estimated UTS in ksi for steels was obtained by dividing the hardness value by two, which was a valuable characteristic. In comparison to previous hardness tests, this characteristic helped it acquire attention early on.



Fig 4.1 Cryotreated Hardness Test Specimens

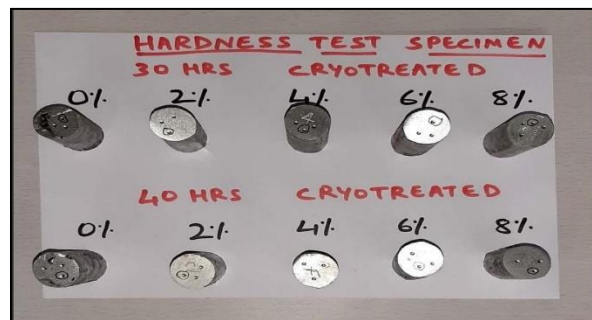


Fig 4.2 Cryotreated Hardness Test Specimens

B. Compression Test

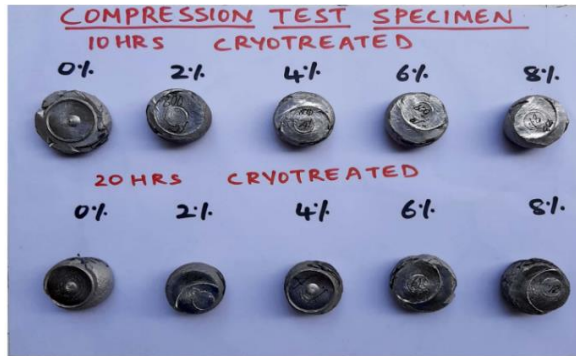


Fig 4.3 Cryotreated Compression Test Specimens

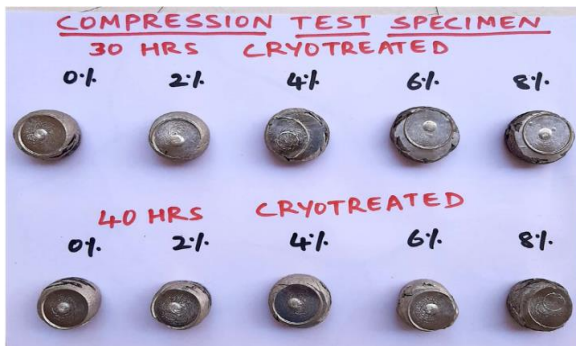


Fig 4.4 Cryotreated Compression Test Specimens

The compression tests were conducted on cast composite with a diameter of 20 mm and a length of 20 mm. Force was applied slowly, then matched strains were evaluated until the cast composite specimen failed.

C. Tensile test

Tensile specimens of diameter 12.5mm and the gauge length 65mm were prepared from cryotreated Castings as shown in Fig. The ultimate tensile strength of the ASTM E8-13a-compliant samples was computed using the observed values from the universal testing equipment, and the results were recorded.

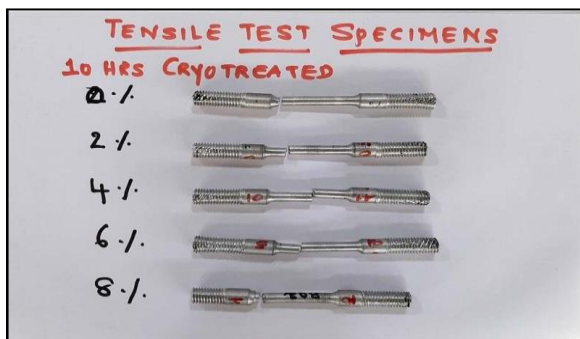


Fig 4.5 Cryotreated Tensile Test Specimens

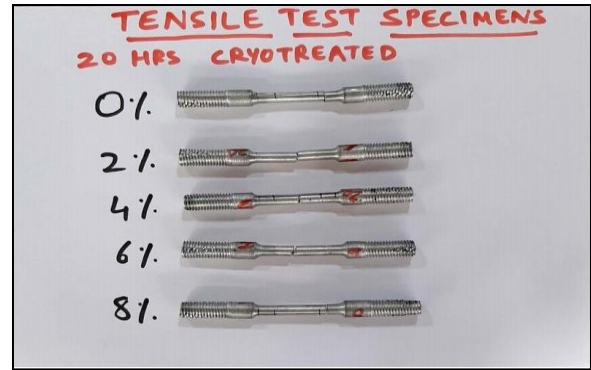


Fig 4.6 Cryotreated Tensile Test Specimens



Fig 4.8 Cryotreated Tensile Test Specimens

D. Scanning Electron Microscopic Studies

The electron column, sample chamber, EDS detector, electronics console, and visual display monitors of a typical SEM apparatus.

VI. RESULTS AND DISCUSSIONS

A. Optical Micrographs

The micrographs of the specimens were analyzed by using Nikon Microscope LV150 with Clemex Image Analyzer with various magnifications (50X, 100X, 200X, 500X). The microstructure varying wt % of reinforcement (2, 4, 6, and 8) was carried out but microstructure for 6 wt% showed the best dispersion structure. Fig. (a-d) shows the micrographs of the composite materials with 6 Wt% with different hours of cryo treatment. Fig (a) comprises fine eutectic silicon scattered in the interdendritic area and fine precipitates of alloying components in the matrix of aluminum solid solution. Fig(c) to Fig (d) Depicts consistent dispersion of ceramic particles with better wettability for Al6061-ZrO<sub>2</sub> MMC. Also, there is an aggregation of particles in composites, Improper stirring might have led to bunch formation of reinforcement particles.

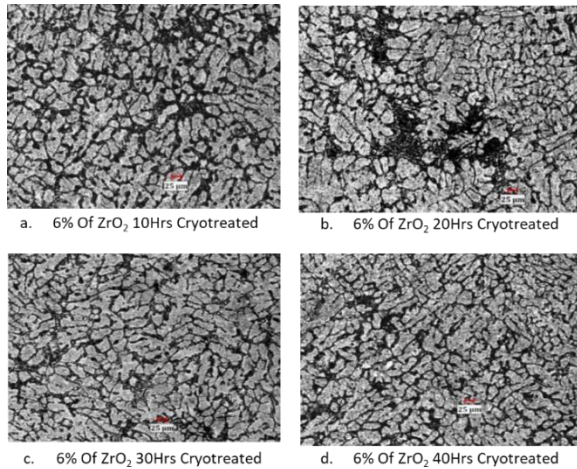


Fig 5.1 (a-d) Micrographs of 6 Wt% of Reinforcement with different hours of Cryotreatment

Length of dendrite structure appeared in Fig (a), decreases in Figs (b-d), which may be due to Reinforcement particles. Zirconium Di-Oxide particulates act like an obstacle to the development of the dendritic structure. Eutectic silicon provinces across particles to a great extent affirmed in micrographs. Eutectic silicon dispersion is more elegant and the structure changed from needle-shaped to spherical around the particles. Porosity is not dominant but cluster and segregation of particles are observed with higher reinforcements. The microstructure for 6% reinforcement with Cryotreated for varying hours is shown, 30hrs of cryo treatment indicated the uniform distribution of reinforcement particulates and due to which enhanced mechanical properties are seen.

B. Hardness Evaluation

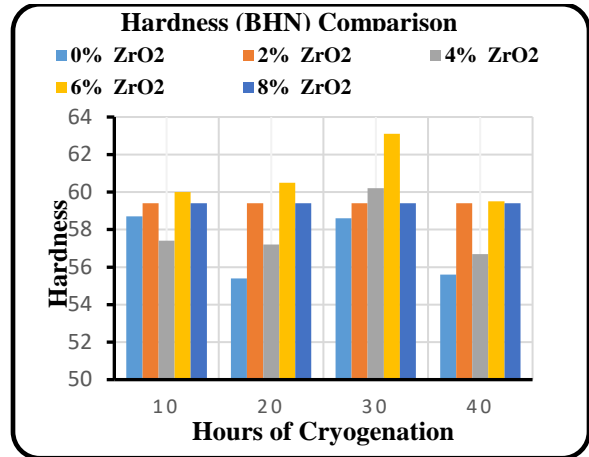


Fig 5.2 Hardness Comparison

There is an appreciable amount of increase in Hardness with increased reinforcement and hours of cryogenation. The cast specimens exhibited a hardness of 68 and specimens with 6 wt% and 30 hours of cryo treatment exhibited the highest compression strength of 63 MPa. This decrease in hardness can be due to cluster formations.

C. Compressive evaluation

The compression specimens were made in accordance with ASTM E9, which covers compression testing of metals like steel and metal alloys. Mechanical parameters like yield strength, Young's Modulus, compressive strength, and stress-strain curve are determined using this test method.

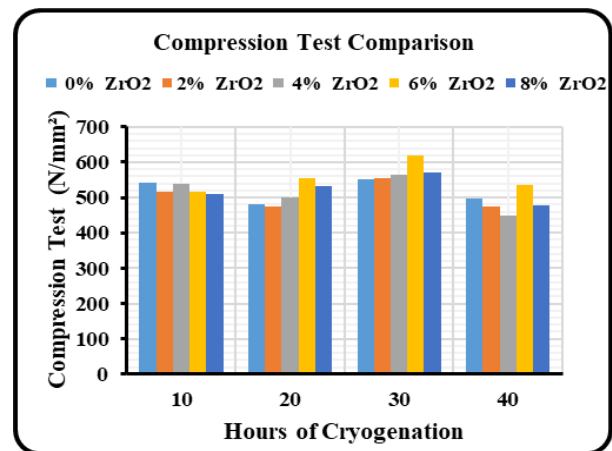


Fig 5.3 Compression Test Comparison

D. Tensile evaluation

The tensile specimens were prepared according to ASTM E8-82 standards and were subjected to uniaxial tensile stresses in a universal testing machine.

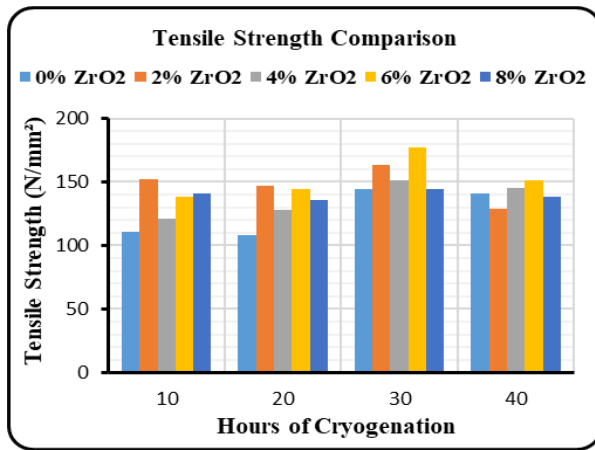


Fig 5.3 Tensile Test Comparison

### CONCLUSION

This research work has highlighted the current research involving Mechanical, and Wear properties of Al6061-ZrO<sub>2</sub> MMCs, which were fabricated by cryogenic technique. The properties of Al alloy MMCs depend on the different variables like dispersion of reinforcement in the base alloy, dispersion of defects, presence of brittle phases in the base alloy, plastic disfigurement of the base alloy and quality at the interphase in an exceptionally complex way. Thus there is a solid requirement for the successful depiction of the attributes of the composites and to extend This research work has highlighted the information pool. Using the stir casting technique, composite castings with Al6061 as the base material and ZrO<sub>2</sub> reinforcement with various mesh sizes of 44 microns (2 percent to 8%) were effectively made. The microstructure examinations revealed a consistent mix of reinforcing particulates up to 8%, after which particle buildup is visible.

The conclusion can be drawn from the investigation carried in the present work that the increase in the percentage of ZrO<sub>2</sub> reinforcements in Al6061 increases the tensile strength, compressive strength, hardness, and fracture strength. ZrO<sub>2</sub> particulates derived from naturally available rock represent an attractive dispersoid to give rise to economical Metal matrix composites. Additions of reinforcement observably increased disruption denseness owing to

changes in CTE. The enhancement in mechanical properties namely hardness, UTS, and compression strength of the composite can be all around ascribed to the prominent disruption density.

It has been found that the Impact quality diminished with increment in % of reinforcement particles. The fracture was ductile with a dimple surface exhibiting particles de-bonding in fracture experiments of produced composites, and particle cracking was caused by transgranular fracture of the ZrO<sub>2</sub> reinforcement.

The ascast specimens exhibited a hardness of 63 and specimens with 6 wt% and 30hours of cryo treatment has a hardness of 68. The composite with 6% ZrO<sub>2</sub> reinforcement shows the highest enhancement in the tensile strength. The % elongation of the composite decreased with the increase in ZrO<sub>2</sub> except in 2% this is mainly due to ZrO<sub>2</sub> is a ductile material.

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