International Journal of Refractory Metals & Hard Materials 75 (2018) 238–247

Contents lists available at ScienceDirect

International Journal of Refractory Metals & Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Mechanical properties of large TiC-Mo-Ni cermet tiles

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ARTICLE INFO

Keywords:
TiC
Cermet
Bending
Toughness
Ultrasound
Microstructure design

ABSTRACT

The mechanical properties of large, commercially produced plates of a 14 vol% nickel bonded $Ti_xMo_{1-x}C$ cermet have been assessed in order to determine their viability for impact and structural applications. Keene et al. (Agnew et al., 2017 [6]) have recently characterized the phase fractions and composition, microstructure (particle size and contiguity), density, and residual stress. Samples tested in the as processed, stress relieved and hot isostatic pressed (HIP) conditions revealed no discernible trends with post-processing. Pulse-echo ultrasonic velocity measurements indicated the Young's and shear elastic moduli of the cermet to be 382 and 155 GPa respectively; consistent with Hill composite estimates. The combination of a moderately low theoretical density of 5520 kg/m^3, combined with a high compressive strength of 2.7 GPa and fracture toughness of 16.1 ± 1.7 MPa$\sqrt{m}$, indicated the material holds promise for some structural applications. However, the low bend strength of only 538 ± 109 MPa, combined a with a low Weibull modulus of approximately 6.3 were indicative of a highly stochastic material that seriously impedes its use in such applications. The random occurrence of large pockets of insufficient binder (a form of shrinkage porosity) between essentially spherical carbide particles contributed to the low Weibull modulus, but did not account for the low average flexural strength. Instead, a high carbide particle contiguity factor was shown to be responsible for the low local toughness and decrease of the critical flaw length to that of cracked carbide particle crack. The structure-property relationships shown to describe the present material were used to construct a graphical Pareto optimization map to examine the strength – toughness relationships of this material system as the hard particle diameter and volume fraction were varied. The development of improved processing approaches that reduce contiguity and porosity are necessary for this material to fulfill its significant potential.

1. Introduction and background

Metal bonded ceramics with high volume fractions of ceramic particles (cermets) have been widely used for metal cutting applications because of their very high hardness, resistance to wear and excellent thermal shock resistance. Cermets have also attracted interest for ballistic impact protection, especially for scenarios where multiple impacts are likely [1–4]. Recent experimental investigations of the impact response of a conventional Al_2O_3 ballistic ceramic and two Ni-bonded TiC cermets showed the single impact ballistic resistance of the cermets to be similar to that of the ceramic [3]. Furthermore, the cermets exhibited less macroscopic radial and cone cracking, rendering them of interest for multiple-hit protection. These materials have also been investigated for making ultrahigh specific strength cellular lattice structures [5]. However, the optimization of each of these cermet applications requires an improved understanding of the relationship between their mechanical properties, their composition/microstructure and the various defects/flaws that might develop during their synthesis and further processing.

A recent study has investigated the microstructure and defect populations of the same (DDG-X grade) Ni-bonded $Ti_xMo_{1-x}C$ cermet investigated in the ballistic impact and lattice structures studies [6]. Image analysis of scanning electron micrographs, quantitative x-ray diffraction (XRD), and micro X-ray computed tomography (μ-XCT) were used for phase volume fraction determination. The three techniques indicated the cermet consisted of 86 vol% Ti(Mo)C ceramic and 14 vol % nickel-molybdenum alloy binder (with an uncertainty of ± 1.25 vol %). The average estimated porosity levels in the as-received material were in the range of 1.6–5 vol% with the lower estimate deduced by surface image analysis and the upper one from μ-XCT analysis. Based upon the measured phase chemistry and volume fractions, the theoretical density of the cermet was calculated to lie between 5410 and 5770 kg/m^3. The significant range in these estimates resulted from the 2.5% uncertainty in the metal composition and the carbon fraction

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https://doi.org/10.1016/j.ijrmhm.2018.05.005
Received 26 February 2018; Received in revised form 30 April 2018; Accepted 7 May 2018
Available online 09 May 2018

(stoichiometry) of the $Ti_xMo_{1-x}C$ phase (large carbon vacancy concentrations are common in such carbides).[1] The measured absolute density (based upon bulk mass and geometry measurements) lay between 5460 and 5560 kg/m^3, indicating a pore volume fraction in the as-synthesized material of 0 to 5.3 vol%, encompassing the range of values obtained by image and μ-XCT analysis, for a range of different samples.

The elastic properties of these composite materials are expected to lie between well-established material property bounds. However, since the volume fraction of the stiff, hard carbide phase is > 80 vol%, the "contiguity" of the microstructure in this percolation regime is likely to control the strength (or hardness) [7,8]. In contrast, the toughness of these cermets is more likely to be dominated by energy dissipation within the metallic phase [9], and would be expected to exceed the intrinsic toughness (1.5 to 4 MPa$\sqrt{m}$) of the carbide phase itself [10].

Here, we measure the elastic moduli, compressive and flexural strength, and the mode I fracture toughness (K_{Ic}) of the same DDG-X grade, TiC-Mo-Ni cermet investigated in the previous study of microstructure and defects [6]. The flexural strength is analyzed in terms of Weibull statistics, porosity distribution and contiguity, while other properties are assessing using bounding models. The implications of the analysis for improved microstructure design are also discussed.

2. Experimental procedures

2.1. Material

Approximately 150 mm × 150 mm square plates of DDG-X grade Ni−Mo bonded TiC cermet were provided by the Davis Defense Group (Fredericksburg, Virginia, USA) with nominal thicknesses of 6, 10, and 12 mm. Their microstructure was characterized in the earlier study [6], and is schematically illustrated in Fig. 1. Briefly, chemical analysis showed the cermet to be composed of 59% Ti, 11% C, 23% Ni, and 7% Mo (all wt%). Energy dispersive x-ray spectroscopy indicated the carbide phase had a metals-basis composition of 88 wt% Ti, 11 wt% Mo and < 1 wt% Ni. Similar analysis of the binder gave a composition of 94 wt% Ni, 5 wt% Ti, and < 1 wt% Mo. The average carbide particle size was ~10 μm and the particles exhibit a core-rim structure with several rims though which oscillating levels of Mo and Ti were observed. Many of the carbide particles were in contact with each other (they had a high contiguity) and regions of missing metal binder were present leading, to the presence of voids (shown as white regions in the figure) that were up to several hundred microns in length.

The as-received tiles were found to be susceptible to fracture during sample preparation, indicative of the presence of residual stresses. This hypothesis was confirmed via measurement of TiC phase residual stress by x-ray diffraction [6]. Some of the as-received cermet tiles were therefore stress-relieved by heat-treating in vacuum at 900 °C for 30 min followed by slow cooling at a rate of 15 °C/min. This was found to be an effective method for eliminating the residual stresses induced by differential cooling through the tile thickness. Some of the as-received material was subjected to containerless hot-isostatic pressing (HIP) in an attempt to reduce the porosity. However, the density of the material was unimproved, and the already high particle contiguity was slightly increased [6]. The contiguity of the carbide particles in cermet systems has been found to be influenced by the carbide particle-liquid metal interfacial energy [11], and a highly contiguous carbide particle morphology has been linked with the presence of low energy interparticle boundaries in both WC/Co [12] and Ti(C,N)/Ni [13] cermets.

2.2. Ultrasonic measurement of elastic moduli

The elastic properties of the TiC–Ni cermets were measured via longitudinal and shear wave pulse-echo ultrasonic techniques. In order to obtain precise velocity measurements, the sample surfaces were polished to a finish of 1 μm, with a thickness variation of < 0.01 mm over the entire length (~30 mm) of the specimen. The average thicknesses of the samples were computed from micrometer measurements at each corner and one at the center of the samples. Samples in both the as-received and HIP conditions were examined and standard error propagation techniques were employed to determine the uncertainty of the measurements. A sinusoidal pulse from a Panametrics 5072PR pulse generator was used to excite Panametrics longitudinal and shear ultrasonic transducers with a center frequency of 2.25 MHz. The transducer waveform was band-pass filtered to minimize noise in the data, and then recorded using a Tektronix TDS 3032C digital oscilloscope. In order to order to determine the time of flight more accurately, an autocorrelation function approach was used [14]. Once the time-of-flight, τ was obtained, the (longitudinal or shear) elastic wave speed, v in the medium was calculated using, $v = \frac{2d}{\tau}$, where d *is* the sample thickness, and τ the time of flight.

A previous study has shown the crystallographic texture of the DDG-X to be random [6], and the material (from Neumann's principle) is therefore elastically isotropic. Thus, the linear elastic stiffness tensor has two independent tensor components C_{11} and C_{44} (or shear modulus, G) that were deduced directly from the sound speed of the longitudinal, v_L, and transverse, v_T, wave velocities as follows:

$$C_{11} = v_L^2\rho;\ C_{44} = G = v_T^2\rho \tag{1}$$

where ρ is the density of the material previously estimated to be 5520 kg/m^3 [6]. The Young's modulus, E and Poisson's ratio, ν of an isotropic solid can be determined from the velocity data in a in a similar way using:

$$E = \frac{v_L^2\rho(1+\nu)(1-2\nu)}{(1-\nu)};\ \nu = \frac{\frac{1}{2} - \left(\frac{v_T}{v_L}\right)^2}{1 - \left(\frac{v_T}{v_L}\right)^2} \tag{2}$$

2.3. Hardness, compression and flexural measurements

A standard Vickers pyramidal diamond indenter was used to determine the hardness of the cermet in the as received and HIP conditions. At least ten indents were made on each sample using a load of 9.8 N (1 kg mass) and a dwell time of 15 s. The compressive stress versus strain response was obtained in accordance with ASTM standard E9-89a. The samples were right cylinders with a diameter of 10 mm and height of 25 mm (the recommended aspect ratio for cemented carbides). Testing was again performed on both the as received and HIP condition material, and both the yield strength and elastic modulus determined using ASTM standards E9-89a and E111, respectively. Each sample was first pre-loaded to 2 kN, then loaded to 120 kN (which for the cross-sectional area used here remained below the yield stress), and then unloaded back to 2 kN. Extensometers were used to measure the strain during this process and the Young's elastic modulus was determined from the unload portion of this test. The extensometer was then removed and the samples were either loaded until nonlinearity in the load-displacement curve was observed or to sample failure. A testing machine compliance factor was determined and used to estimate the actual stress-strain curve for the cermet from this load – displacement data. Flexural strength testing was performed in accordance with ASTM standard C1161 for Flexural Strength of Advanced Ceramics at Ambient Temperature. The samples were rectangular bend beams, with nominal dimensions of 8 mm wide × 48 or 52.5 mm long × 3.5 mm thick. All samples were tested in four-point bending in which the outer span length (40 mm) was twice that of the inner span (20 mm). Load-deflection data was collected for the specimens as the samples are loaded until failure. The flexural strength, *FS* was computed using:

[1] The C stoichiometry of the carbide was assumed to lie between 0.8 and 1.