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INVESTIGATION OF THE MECHANICAL BEHAVIOUR OF METAL-DIAMOND COMPOSITES

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Abstract
Metal-Diamond Composites (Me-CD) are a novel class of materials which has typical applications in the field of thermal management. Usually, due to the high volume fraction of diamonds inside the matrix, the mechanical behavior of such materials is quite brittle with low level of fracture stress and strain. However, with advanced innovations in the sintering processes, it is possible to obtain composite materials with a good level of strength and toughness. The great advantage of these materials is the possibility to combine the high thermal and electrical conductivity of diamonds with the strength of metals.
Aim of this work is the investigation of the mechanical behavior of Me-CD from quasi-static to high strain-rate loading conditions. The temperature influence on mechanical properties is also evaluated.

1 Introduction
Metal Diamond Composites (Me-CD) are a new class of metal matrix composites that are exiting the interest of designers and engineers because of their unique combination of physical, thermal, electrical and mechanical properties. Fields of interest are all situations where a high thermal stability is needed (see the thermal stability index and the thermal shock resistance reported in Table 1): heat exchangers, heat coolers for CPUs, etc.
In general, due to the high volume fraction of diamonds inside the matrix, the mechanical behavior of such materials is always brittle with low level of fracture stress and strain. However, with advanced innovations in the sintering processes, it is possible to obtain composite materials with a good level of strength and toughness even in high strain-rate conditions. The great advantage of these materials is the possibility to combine the high thermal and electrical conductivities of diamonds with the strength of metals. The use of refractory metals, which have a high melting point and elevated thermal stability, leads to an ideal thermal management material that maintains its thermo-mechanical properties up to very high temperatures.
Thanks to these combinations of properties Me-CD could find several applications in particle accelerators and nuclear-related technologies, where problems of thermal management are combined with structural requirements and resistance to radioactive environments. However, this class of materials could also be used in every case in which the material requirements are

hardness combined with ductility and lightness (e.g. in military protection devices instead of alumina or carbides).

Due to the composite nature, where the diamond particles are bonded to the metallic matrix, the strain and stress distribution in the material is quite complex and, as a consequence, the mechanical behavior of the material is different from a homogeneous one.

Aim of this work is the investigation of the mechanical behavior of Me-CD from quasi-static to high strain-rate loading conditions. A series of mechanical tests (at different temperatures and strain-rates) was performed in order to understand the intrinsic failure mechanisms of this new family of advanced materials.

2 Molybdenum-Diamond Composite

The most known metal matrix composite is Copper Diamond (Cu-CD): Cu shows a very high thermal and electrical conductivity that is reflected in the Cu-CD properties. However, the low melting point of Cu and the poor interfacial bonding between copper and diamond are seriously limiting the application of this material. Cu-CD is a very good conductor but is intrinsically brittle and has a low thermal stability at high temperatures [1].

For those reasons, to find a better compromise for the new Phase II LHC Collimators an R&D program [2] has been established to produce a Metal Diamond Composite based on molybdenum instead of copper. Molybdenum-Diamond (Mo-CD) has been developed inside the collaboration between CERN (EN/MME group) and BrevettiBizz (Verona, ITALY), a private Italian company, during 2010-2011.

Molybdenum is a refractory metal that exhibits the high thermo-mechanical properties and keeps them up to very high temperatures thanks to its high melting point (2623 °C). The objective of the R&D program is to obtain a composite material possessing good thermal properties (higher than pure Mo but obviously lower than other metal diamond composites like Cu-CD) in addiction to good mechanical properties and tailored density.

	CFC	**Mo**	**Glidcop**	**Cu-CD**
Density (kg/m3)	1650	10220	9800	5330
T_{melting} (°C)	3650	2623	1083	1083
Thermal Conductivity (W/m/K)	60	140	365	490
CTE (10^{-6} K^{-1})	1.5	5.3	16	8-13
Young's modulus (GPa)	80	320	130	210
Thermal stability index: k/α	40	26	23	75
Thermal shock resistance: $\frac{\sigma_y (1-\nu) C_p}{E\alpha}$	793	55	38	17
Electrical conductivity (MS/m)	0.143	19.2	54	12.6

Table 1: Comparison of properties of interest and parameter indexes at RT for some collimator related materials.

Furthermore, Mo forms stable carbides on diamonds surfaces during the process (unlike Cu), which provide the necessary bonding strength to the composite. Since the carbides have low

thermal conductivity and high brittleness, the carbide layer must as thin and homogeneous as possible (~1 micron).
The R&D program of Mo-CD led to the identification of the composition and process parameters allowing the best compromise between thermal and mechanical properties: five plates having dimensions 200×80×4 mm have been produced and extensively tested at CERN, Politecnico di Torino and, for future radiation hardness characterization, RRC-Kurchatov Institute. In this work only the mechanical tests performed at CERN and Politecnico di Torino are described.
Mo-CD is obtained by Rapid Hot Pressing (RHP) like Cu-CD, but since the temperature needed to properly sinter molybdenum is much higher than in Cu-CD sintering (due to the higher melting point of Mo), diamond degradation poses a major challenge in Mo-CD production.
To reduce the temperature to an acceptable level (1100-1300°C) a Liquid Phase Sintering (LPS) process has been explored. A third low-melting phase (Cu) is added to the mixture to fill the pores left by Mo and CD particles. The presence of Cu must be kept to a minimum to reduce the effect of the low thermal stability of Cu. As a matter of fact, in Mo-CD copper acts only as a filling element, while the skeleton of Mo interconnected particles bonded to the diamonds by the Mo_2C interface are giving the mechanical strength to the composite.

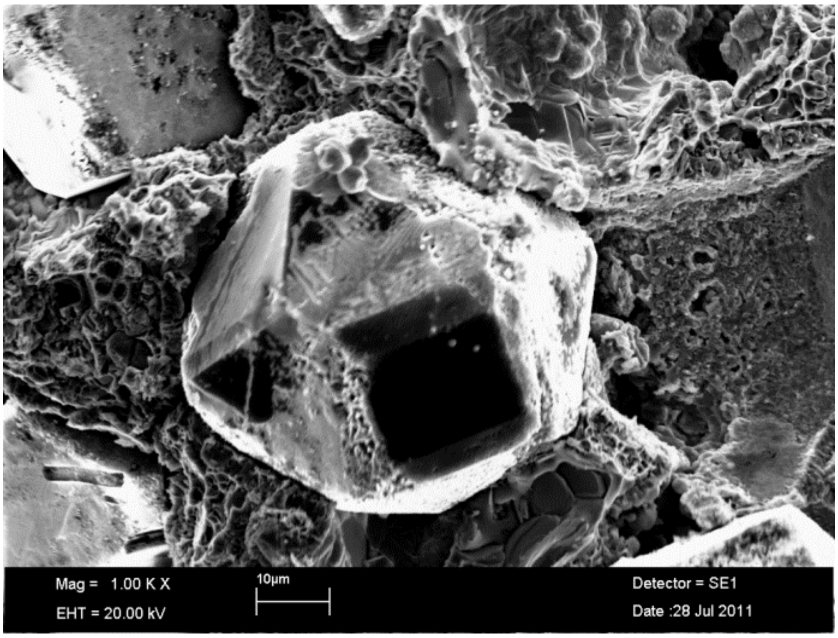


Figure 2 – SEM microstructure of Mo-CD fracture surface. This passes through the Mo_2C interface between CD and Mo-Cu matrix. Some Mo particles are still attached to the diamond, which is not degraded and maintains good regularity of the surfaces.

Usually, the fracture surface in Mo-CD passes through the interface between diamonds and Mo-Cu matrix, which is made essentially by Mo_2C carbide formed during sintering. This situation can be clearly seen in Figure 2 and 3.
Mo_2C is intrinsically brittle. However the final composite material shows good mechanical properties: this fact can be explained looking at the disposition of the Mo particles surrounding a diamond, as presented in Figure 4.
During the sintering, large diamonds are surrounded by liquid Cu and small solid Mo particles: due to the applied pressure and the mobility of Cu, Mo tends to migrate onto the diamond surface forming Mo_2C carbide. The result is a ~ 5 micron thick Mo coating (connected through the intermediate layer of Mo_2C) that completely surrounding the

diamonds (Figure 4). This structure provides an optimal bonding between the Mo-Cu matrix and the diamonds, leading very good mechanical strength, as confirmed by the experimental results.

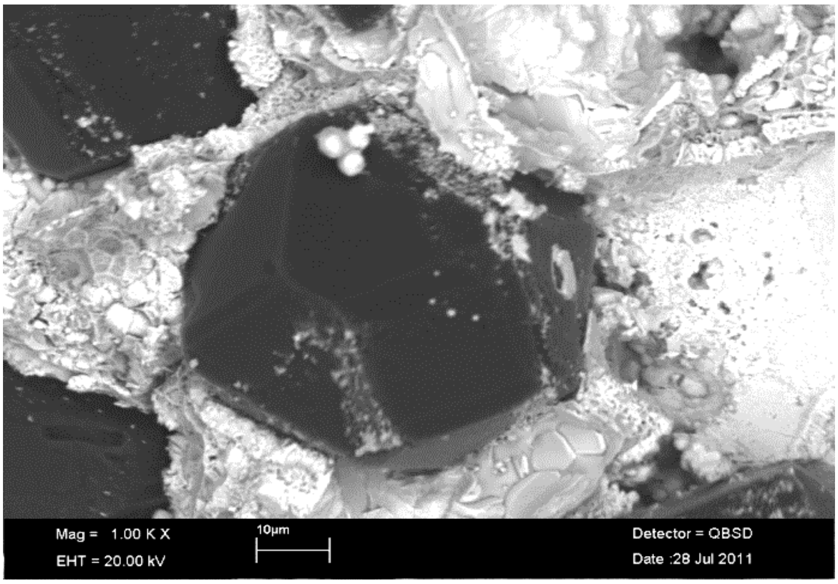


Figure 3. Same picture as Figure 2 using Back Scattered Electrons (enhancing differences in atomic weight). The grey areas on the diamond surface indicate the presence of Mo_2C at the interface.

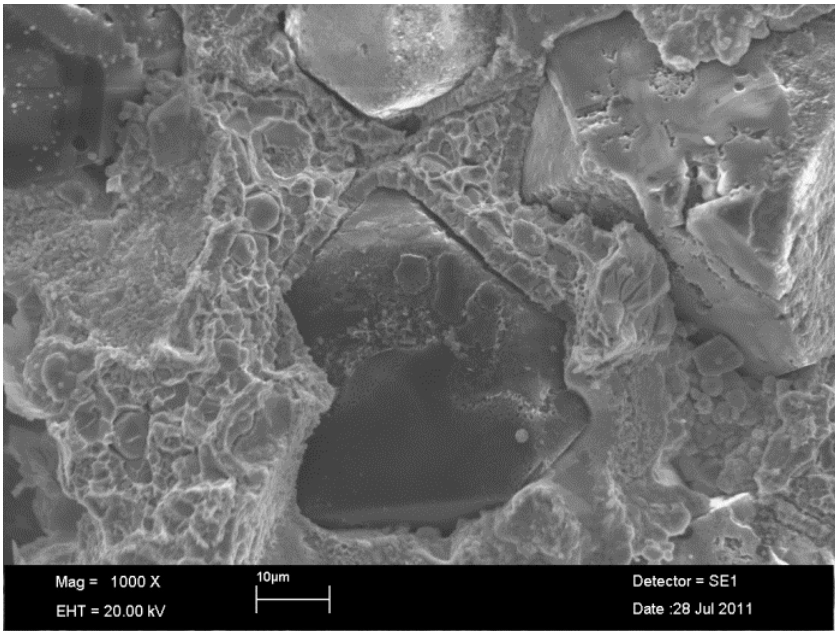


Figure 4 - SEM microstructure of Mo-CD. The small Mo particles surround the diamond particles, forming a regular coating on the latter. The small interstitials between the Mo particles have been filled by melted Cu.

3 Mechanical tests

It is quite difficult to obtain samples from the sintered plate and a water jet cutting is necessary since the high hardness of the material. A first evaluation of mechanical behavior of the material and the analysis of the strain-rate influence was investigated by compression tests covering six orders of magnitude in strain-rate on small cylindrical samples (5 or 6 mm of diameter and 4 mm of length). Due to the brittle nature of Me-CD, there is a significant dispersion of the experimental results. For this reason, a great number of repetitions were done for each testing condition. Moreover, the material is realized starting from powders: this causes non-uniformity in the spatial distribution of the particles, influencing the composition. This aspect is investigated evaluating the electrical conductivity in several points on each plate of Mo-CD, as reported in figure 5.

Figure 5. 200×80×4 mm plate of MoCD. The plate has been checked with SigmaTest instrument to verify the local homogeneity of Electrical Conductivity (in MS/m).

The experimental tests were performed starting from quasi-static loading conditions up to high dynamic ones. All the experimental tests were performed in the Reliability and Safety Laboratory of the Politecnico di Torino (Vercelli).

The low-speed tests (at about 10^{-2} s^{-1} strain-rate) were performed with a general purpose electro-mechanical material testing machine, Zwick Z100. This equipment is able to apply loads up to 100 kN, measured by a 100 kN load cell, at a speed of up to 5 mm/s.

Dynamic tests were performed by means of a standard Split Hopkinson Pressure Bar (SHPB) setup (Fig. 6). The apparatus was actuated by a pneumatic gas-gun (1.5 m long) and was composed of two bars made in high strength steel of 10 mm diameter and 3.4 m length. The striker bar used in these tests was 500 mm long: with this setup the impact velocity is about 20 m/s. This implies that with the adopted specimen diameter (which determines the ratio between transmitted and incident waves) and lengths (which determine the strain-rate once the reflected wave has been measured), the actual strain-rates varied between 1000 s^{-1} and 2000 s^{-1}. Strain measurements in the bars were performed with KYOWA semiconductor strain-gages. Acquisition was made with a 2.5 MHz NI acquisition board managed by a LabView program.

Figure 6. SHPB setup used for high dynamic tests of the Mo-CD samples.

An algorithm for the wave dispersion correction in the bars is necessary to obtain a good level of accuracy in small strain measurements [3]. To reduce the variation of strain-rate due to dispersion, an adequate pull-shaper was adopted: this allows having a more precise achievement of the equilibrium condition in the specimen.
In Figure 7 the maximum strength values obtained from the quasi-static and SHPB tests are reported for all the compression tests performed. The data dispersion is quite evident and it is possible to conclude that the material does not show a significant strength increase as consequence of the rise in strain-rate.

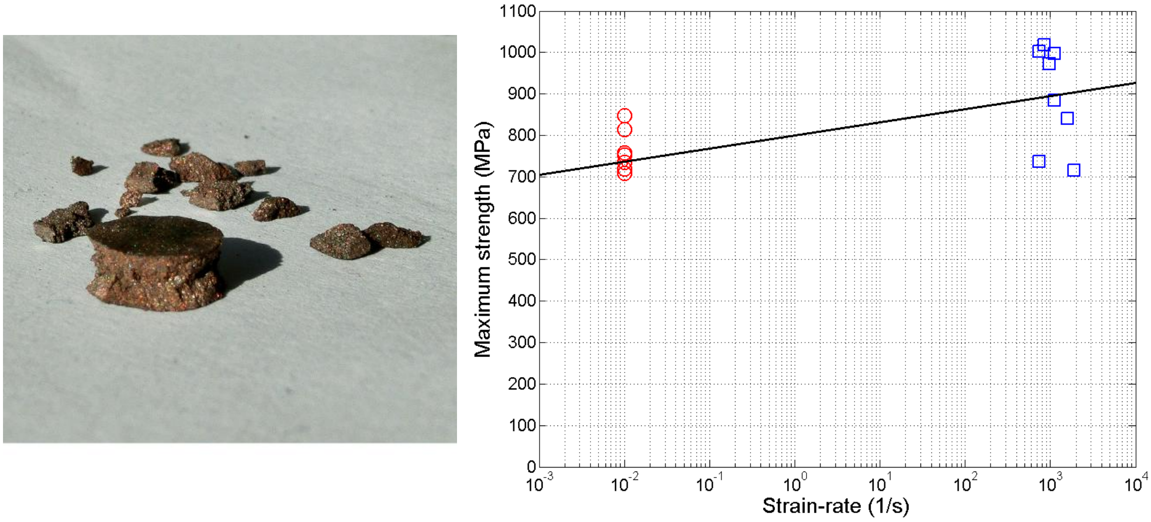
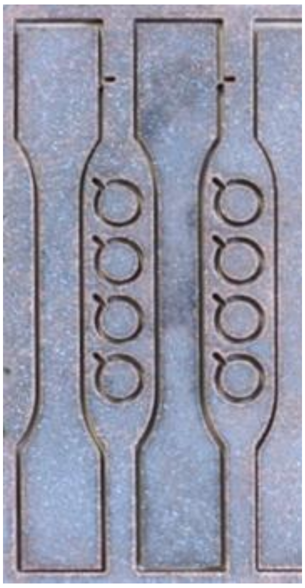


Figure 7. Picture of the cylindrical compression specimens used for quasi-static and high strain-rate tests (left); results in terms of maximum strength vs. strain-rate (right).

In a second phase, also the behavior in tension was investigated. Dog-bone specimens (gauge length 60 mm, width 10 mm and thickness 4 mm) were obtained by water-jet cutting, see figure 8. The material was tested at quasi-static loading rate from room temperature up to 600 °C. To reach the desired temperature an induction heating system was adopted and controlled in a feedback closed-loop with thermocouple welded on the specimen surface. The engineering stress-strain curves are reported in figure 8. The strain was measured by an extensometer, and this allows estimating with a good level of accuracy the Young's modulus at the different temperatures. The results show that the material exhibits a little phase of plasticity before the fracture. The significant reduction of the stress level and elastic modulus that occurs a high temperature could be due to the presence of the phase with a low melting point (Cu).



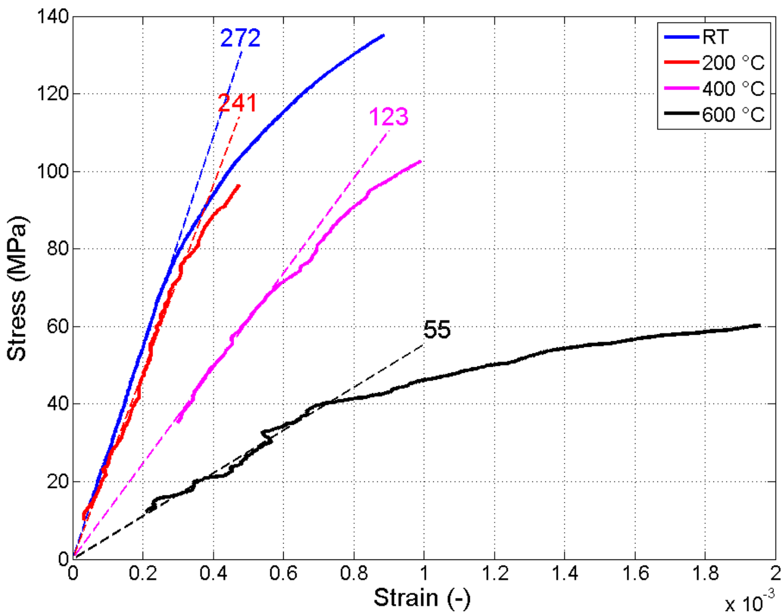
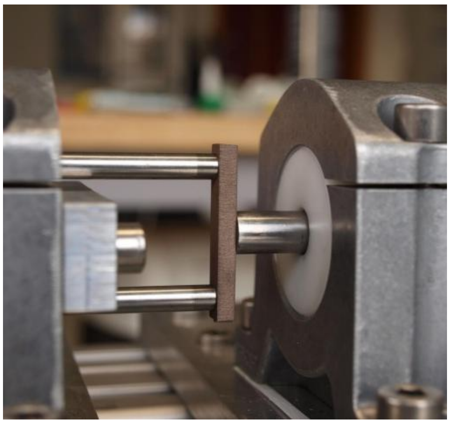


Figure 8. Picture of the dog-bone specimens used for quasi-static tests at different temperatures (left); results in terms of engineering stress-strain curves and evaluation of the elastic modulus (right).

Unfortunately, because of the difficulties in the realization of specimens compatible with the Hopkinson setup in tension (setup available in the laboratory of the Politecnico di Torino), it was not possible to perform tensile tests at high strain-rate. In order to try to compensate for this lack, the bending setup for the Hopkinson bar was developed [4]. The choice of this type of tests is justified since at CERN laboratory quasi-static bending tests are usually performed for evaluating the mechanical properties. As it is well known, bending tests produce a non-uniform stress state inside the specimen: this makes the results non universal and strongly dependent on both the geometry and the behavior (brittle or ductile) of the material. However, the bending test can be useful for the comparison of different materials or the same material with varying loading rate. In figure 9 the picture of the setup and the results in terms of maximum strength are reported. As for compressive tests, the data dispersion is quite evident and again there is a little influence of the strain-rate on the mechanical response of the material. The maximum strength was evaluated in both dynamic and static tests with an elastic distribution of the stress in the sample beam in a 4 points bending loading condition.



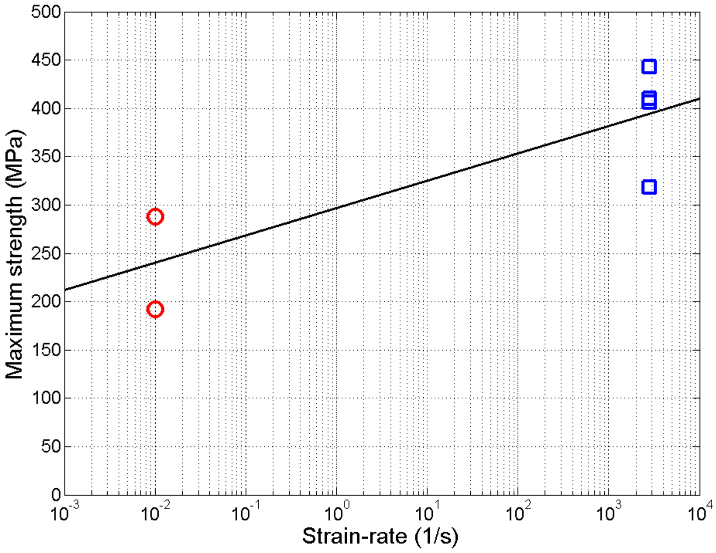


Figure 9. Picture of the experimental setup for the Hopkinson bending tests (left); results in terms of maximum strength level vs. strain-rate (right).

4 Conclusions

In this work the mechanical response of metal diamond composite was investigated. In particular, the attention was focused on the molybdenum diamond composite developed at CERN in collaboration with the Italian company BrevettiBizz. Compression tests were performed from quasi-static up to high strain-rate loading conditions. The dynamic tests were performed via a standard Split Hopkinson Pressure Bar. The obtained results show a significant dispersion of the data and a little influence of the loading rate on the maximum strength level. Tensile tests were performed in quasi-static regime at different temperatures (from room temperature up to 600 °C). The evaluation of the Young's modulus was performed using an extensometer for the measuring of the strain. A significant reduction of the strength was observed at the maximum temperature. Probably this is due to the presence of the copper (low melting point) in the material. Finally, bending tests on the Hopkinson bar were performed and the results compared with the result obtained from quasi-static tests. The experimental data are quite dispersed and do not indicate a significant strain-rate sensitivity of the material.

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