Fracture toughness measurements of powder metallurgical (P/M) green compacts: A novel method of sample preparation

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The fracture toughness of unsintered powder compacts has, until recent times, been of little interest to the powder metallurgical community. The introduction of new compaction technologies, such as metal injection molding, double pressing and warm compaction, all of which enable high density/high strength green parts to be produced, has changed this situation. High strength powder compacts offer opportunities for techniques such as green machining to be exploited. However, without extensive knowledge of the mechanical behavior of the compacts, including fracture toughness, it difficult for designers and engineers to predict the response to external forces during the machining process. A wide variety of techniques are available to determine the fracture toughness of brittle materials. These can be broadly split into two groups; those which employ conventional fracture mechanics using notches and secondarily induced precracks; and those based on a sharp indenter approach that introduce diagnostic microcracks by Vickers or Knoop indentation. In the former category, traditional methods include single-edge notched beam (SENB) [1], single-edge precracked beam (SEPB) [2] and chevron-notched beam (CNB) [3] techniques. In the past, no particular preference has been given to any of these methods for determining the fracture toughness of brittle specimens because each of them has appreciable shortcomings, which restrict their application. Recently, however, much attention has been focussed on the single-edge V-notched beam (SEVNB) [4] method, which is a further development of the SENB method, as a reliable technique to accurately assess the fracture toughness of brittle materials. This method usually involves sharpening the tip of a SENB saw cut notch by using either a razor blade sprinkled with diamond paste or very specialized micro-machining techniques. In this way the stress concentration at the bottom of the notch is enhanced, leading to a sharp crack front during subsequent testing [4].

The fracture toughness testing of green powder compacts is significantly more problematic than the testing of most other brittle materials, such as ceramics, and cannot be approached by conventional means. For instance, introduction of a precrack into a compacted powder test bar by fatigue would prove extremely difficult given the low strength (compared with the sintered equivalent) and the presence of essentially random porosity (which in many cases is interconnected). In light of this, methods which involve mechanically notching the green specimen must be employed. In the authors' experience however, mechanically machining any sort of notch (even with a razor blade) inevitably leads to mechanical damage to the notch in the form of particle pull out and entrapment of debris in the pores. During subsequent fracture toughness testing, the crack front may initiate from this damaged material away which is unacceptable if reliable and accurate fracture toughness values are to be obtained. The same sort of arguments prohibit the use of sharp indenter type methods in which pore collapse beneath the indenter would accommodate most of the strain induced and preclude the production of measurable, radial cracks.

Another method which has been employed successfully to introduce notches into sintered materials is electrical discharge machining (EDM). This technique can cut a notch approximately 200 μm wide to any depth after which razor blade V-notching can be performed at the notch tip if required. Unfortunately, EDM notching cannot be employed to notch green powder compacts since the technique involves the use of an aqueous electrolyte which, in most cases, infiltrates the compact and leads to problems such as particle decohesion, corrosion and binder degradation. The EDM process also introduces the possibility of metallurgical modification to the surrounding microstructure.

We propose in this paper a simple method of introducing a sharp V-type notch into green compacts without inducing any damage or residual stresses to the locality of the notch tip. The notch root radius is an important feature of the V-notch and should obviously be as small as possible if the "true" fracture toughness is to be measured. It is generally agreed that, on a practical level at least, notches introduced by a razor blade are the best solution. In the proposed method, sharp notches are made by compacting powder around a razor blade rather than using the blade to cut notches, as is the general practice. In this way blunting of the razor blade during the cutting action itself is also avoided. Compaction is performed using conventional punch and die equipment, the bottom punch of which has been modified to accommodate a razor blade edge. In this work, a cylindrical die (22.3 mm diameter) has been used and fracture toughness measured using the single-edge V-notch (SEVN) diametrical compression method [5]. A 2 mm deep, 300 μm wide notch was cut, using EDM, across the centerline on the surface of the bottom punch. Using a diamond cut off wheel, a 3.6 mm wide × 22.3 mm long section was removed

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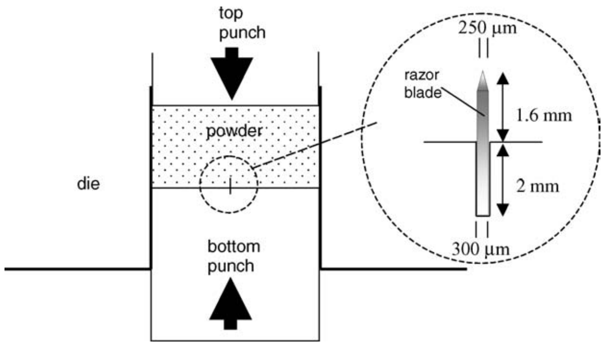


Figure 1 Schematic diagram of procedure used to produce SEVN diametrical compression specimens.

from a 250 μm thick razor blade (Agar Scientific) and inserted into the groove on the punch surface. This resulted in a 1.6 mm of razor edge protruding above the surface which subsequently would form the notch in the compacted specimen. Fig. 1 shows schematically the experimental set-up used to produce cylindrical specimens for subsequent SEVN diametrical compression fracture toughness testing.

In this study Distaloy AE densmix powder was used to validate the applicability of the notching method to the fracture toughness testing of green compacts. This powder is a proprietary blend of ferrous-based powder, carbon and lubricant produced by Hoganäs AB, Sweden especially for warm compaction applications. Notched specimens were produced by warm compacting at 130 °C using a range of pressures from 390 to 750 MPa. The height of the pressed specimens was controlled by varying the quantity powder in the die and was aimed at producing specimens in which the notch depth was around a quarter of the specimen height regardless of the compaction pressure and compact density. Fig. 2 shows two views of a typical notch produced in the Distaloy AE densmix powder compacts by the method outlined. Cross sectioning was performed using EDM (to ensure no mechanical damage) and hence, to some extent, the powder microstructure and pore structure has been obscured by corrosion products. However, it can still be seen that the notch is extremely sharp with a root radius in the order of 2–3 μm. Even if a degree of pore closure does occur around the locality of this notch tip during compaction, it will be small in comparison with the plastic zone generated at the crack tip during testing which has been estimated, from mechanical property measurements, to be around 100 μm.

The diametrical compression test involved loading the cylindrical specimens across their diameter such that tensile stresses are created perpendicular to the load. The notch is aligned parallel to the loading axis. The test was performed using a fairly high crosshead speed of 1 mm min^{-1} in order to minimize time-dependent subcritical crack growth. Fig. 3 shows an example of a notched specimen just prior to the commencement of compression together with a typical specimen fracture surface exposed after completion of the test.

Fracture toughness was calculated as follows:

$$K_{\mathrm{IC}} = \frac{2.24}{\sqrt{\pi}} \frac{P_{\mathrm{f}}\sqrt{c}}{DT} \tag{1}$$

where P_{f} is the failure load, c is the crack depth, D is the specimen diameter, and T is the specimen thickness.

The results of the SEVN diametrical compression fracture toughness tests are shown in Fig. 4. Each data point is derived from the average of 5 tests which have a standard deviation, in all cases, of less than 0.03 MPa $\sqrt{\mathrm{m}}$.

It is clear from Fig. 4 that the method outlined is sensitive enough to discern trends in the fracture toughness for this class of green compacts and, given the low

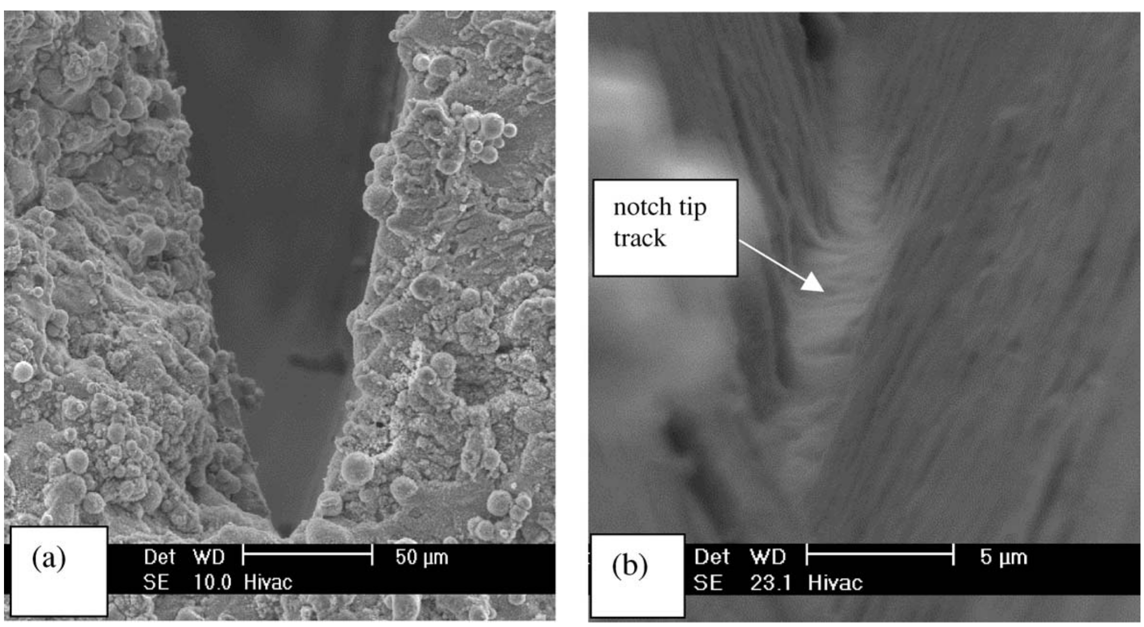
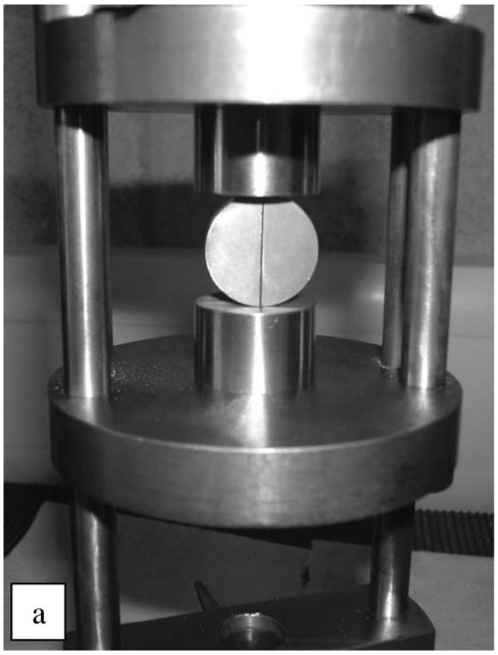


Figure 2 Electron photomicrographs of a typical notch generated in a Distaloy AE densmix compact. Micrograph (a) shows a cross section through the notch while (b) shows a view looking into the notch from above. It should be noted that in (a) the microstructure surrounding the notch has been modified during EDM sectioning.



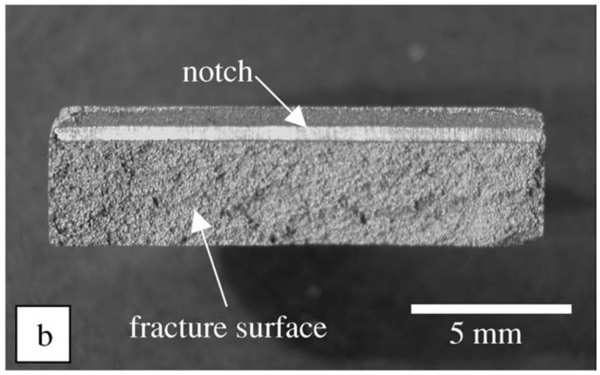


Figure 3 SEVN diametrical compression fracture toughness test: (a) notched specimen just prior to testing and (b) resultant specimen fracture surface.

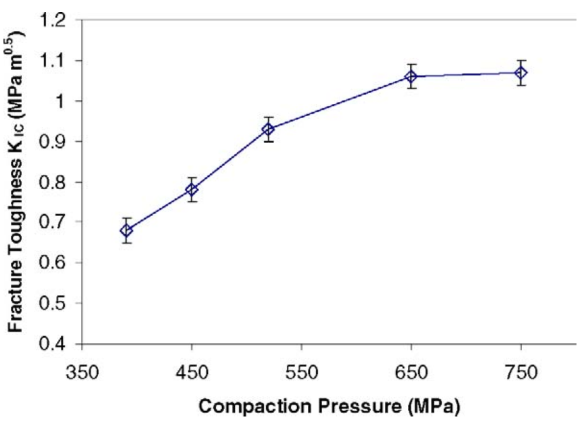


Figure 4 Fracture toughness of warm compacted Distaloy AE densmix specimens plotted as a function of compaction pressure.

standard deviation of the data, provides results with a high level of reliability and consistency. The absolute K_{IC} values obtained seem reasonable and are comparable to values reported for porous ceramics [5].

In conclusion, an innovative and reliable method of notching green powder compacts for fracture toughness testing has been developed. Such a method has potential applications over a wide range of test geometries and powder systems.

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