Brazilian disc experiments on a cold spray material

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Abstract. A series of experiments were performed to probe the tensile behaviour of a cold sprayed reactive metal composite material containing a mixture of nickel and aluminium. Data were acquired at two different strain rates and were collected using high speed photography, strain gauges, force-extension measurements and were analysed using digital image correlation techniques. Comparisons were made with modelling on representative microstructural elements in the CTH code, supporting the conclusion that the material failed in shear rather than tension, in a manner dictated by the microstructure. Fairly high sample to sample variation was observed as well as minimal evidence of strain rate dependent behaviour.

1. Introduction
The field of novel energetic materials, specifically in this case reactive metallic formulations, is one that has increased in prominence over recent years. Many different processing techniques, such as cold spray, swaging, cold/hot pressing and explosive compaction (see [1–3]), and applications for such materials have been suggested (e.g. [4]). One of the attractive features of such materials is that they allow for materials that would normally be inert to be made reactive. An example of this in a system would be in munitions casings [5], which traditionally have only imparted action on a target through momentum transfer. A reactive material would also allow for energy being produced in an exothermic chemical reaction to be deposited to a target. While the attractiveness of reactive fragments is easy to see, it is also important to realise that any reactive casing material has to fulfil all of the other design criteria. The casing has to be able to withstand handling, launching (if required) and so on. This aspect of the material characterisation of reactive metal systems appears to be under-represented in the literature; while there are plenty of shock loading and reactivity studies which explore the ultimate use of the materials, there is a lack of low and intermediate strain rate data. This paper presents one example of such an investigation. Although the likely strengths of reactive metallic materials cannot be generalised owing to the very different material properties and processing techniques (which will strongly affect microstructure) it is possible that the observed mode of failure may be common. The investigation uses Brazilian disc testing [6], which gives an indication of the tensile strength of the material whilst the loading itself is carried out in a compressive fashion. The method is likely to give significantly different results between the sprayed metallic composites and the raw metallic materials making it an important loading geometry for analysing material suitability.
2. Experimental
The materials for this investigation consisted of gas dynamic cold sprayed [7] nickel-aluminium composites. The material was derived from a nickel coated aluminum powder sprayed onto a metallic substrate in an argon atmosphere. The samples were deposited onto a rotating mandrel. The original powder was produced for the Army Research Laboratory by Federal Technology Group of Bozeman, MT USA, and consisted of micron scale components (although the exact particle size distribution was unavailable. The cold sprayed material had the following average properties: \( V_v = 0.51 \pm 0.02 \) Ni, Surface area per unit volume \( S_v = 186 \pm 6 \text{ mm}^{-1} \), as sprayed density \( 5.27 \pm 0.04 \text{ g/cm}^3 \), and the original powder average density was \( 5.55 \pm 0.01 \text{ g/cm}^3 \) based on pycnometer measurements following the procedure outlined in ASTM C135-96 [10]. To check for potential material anisotropy \( V_v \) and \( S_v \) measurements were performed on different planes using the method detailed by Gokhale and Drury [11] and they were found to have overlapping confidence intervals for \( V_v \) measurements. For example 24 measurements each on 3 planes of the same sample yielded \( V_v = 0.46-0.56, 0.52-0.61, 0.50-0.56 \). \( S_v \) measurements often but not always have overlapping confidence intervals (on 3 planes of the same material 164-191,209-231,177-195). The data suggests that the material may be transversely isotropic (i.e. perpendicular to the xy plane in figure 2) but only weakly so if at all. As cold spraying is likely to induce some level of statistical sample to sample variation, two separate rods of material were provided that were subsequently machined into experimental samples 10 mm diameter and 3 mm thick. The samples were split into two batches so that they could be examined under two different strain-rate regimes. Low strain rate testing was conducted on a screw driven Instron testing machine with a crosshead speed of 0.5 mm min\(^{-1}\). Higher strain rate experiments were carried out on a split Hopkinson pressure bar system with Vasco Pacific 350 Maraging steel bars and striker velocities in the range of 7.7-8.2 m s\(^{-1}\). The geometry was a typical one for Brazilian disc tests (see for example Williamson [8]) and involved using curved anvils to reduce the risk of Hertzian cracks forming at the contact points). In addition to the normal diagnostics (force and extension on the Instron and strain gauges on the SHPB) both sets of samples were sprayed with a random speckle pattern (maximum speckle size was on the order of 0.1 mm) to allow for the use of digital image cross correlation techniques to measure the strain fields as the samples underwent deformation. It was felt that the presence of a thin paint layer on the surface of the sample was unlikely to affect the results as the strength of the paint layer is negligible in comparison to that of the sample.

3. Results and Discussion
Force-displacement data for the low strain rate experiments can be seen in figure 1 and the failure forces are additionally tabulated in table 1. The offset seen at the beginning of the traces is due to slight initial misalignment of the sample. There is a wide spread of failure forces recorded across the series of samples, indicating that, at least for this experimental configuration, the materials are susceptible to sample to sample variation. The variation is to be expected, the cold spray method involves the compaction of particles fired from a nozzle and does not allow for accurate reproduction of the exact microstructure. Such variation is an important issue for the utilisation of these materials in real world situations as it means, for example, that components would have to be designed with a large safety factor to account for the random nature of the failure (obviously many failure mechanisms have a statistical nature, however the relative variation in this case is relatively large). There is a slight difference between the two rods in that on average rod 1 requires a higher breaking force \((4860 \pm 190 \text{ N})\) than rod 2 \((4170 \pm 220 \text{ N})\), which is in this case also attributed to the random nature of the manufacturing method. In both the higher and lower strain rates used in this investigation failure in the images was taken to be the point at which a visible crack appeared at the observed surface of the specimen. While it is possible that there is some level of internal failure prior to this it was felt that the likely velocity
of crack propagation would mean that the external crack would become visible in a very short space of time. The abrupt appearance of a macroscopic crack is observed between successive frames in SHPB experiments. Based on the camera interframe time we estimate a lower bound for the crack velocity is 400 m/s. There is unlikely to be much strain rate dependence of crack propagation speed.

Digital image correlation performed on the speckle patterns gave consistent strain information in the low strain rate experiments. A sample of the data can be seen in figure 2 which shows the strain data maps and figure 3 which shows the shear data maps for the xy component of shear. The strain fields (showing strain in the y direction, with the loading being in the x direction) from the speckle data show that the material deforms symmetrically for the majority of the experiment. Ultimately the failure of the material has a shear nature, in that the crack is not aligned vertically. Failure in shear is likely because failure originates at the grain boundaries, which are unlikely to be directly in tension. The grain boundaries will localise the shearing, which would possibly account for the inability of the DIC to resolve it. The traditional failure mode in the Brazilian test is a crack that forms vertically, i.e. parallel to the loading axis. In the images showing the shear strain, there is very little shear until just prior to failure, and in particular there is no shear deformation in the centre of the sample where the failure occurs. This would suggest that the shear failure is due to the microstructure of the samples as opposed to a macroscopic experimental consideration such as misalignment of the loading axis. As the failure occurs in shear it is unlikely that any useful conclusions can be drawn from applying standard tensile failure equations (as might be traditionally used in the analysis of Brazilian tests, e.g. [6]) to these experiments and therefore the data have been shown in terms of the failure forces.

![Intron Brazilian Tests](image)

**Figure 1.** Collected data from the low strain rate experiments (Instron cross-head speed of 0.5 mm min⁻¹). While the overall shape of the force-compression curves are similar, on average the failure force for samples from rod 1 is higher than that for rod 2 and there is also significant sample to sample variation within the two subsets.
Figure 2. Strain (y) evolution in Brazilian Test conducted in the Instron at a crosshead speed of 0.5 mm min$^{-1}$. The compression is from top to bottom in the images and the individual images have a scale of 10mm. The colour bar shows strain from $-5 \times 10^{-3}$ to $20 \times 10^{-3}$. The strain evolves symmetrically, but ultimately the failure is in shear at an angle.

Figure 3. Shear (xy) evolution in Brazilian Test conducted in the Instron at a crosshead speed of 0.5 mm min$^{-1}$. The images are equivalent points in the experiment to those in figure 2. Again the compression is from top to bottom in the images and the scales are identical. There is very little shear strain in the samples until the final image, which is post failure.

The high strain rate (Hopkinson Bar) data was more complex to interpret than the Instron data. Oscillations in the output bar trace made it difficult to determine when failure was occurring from the trace alone. In contrast, the low strain rate data showed a continuous rise to a well defined failure. In order to determine which of the oscillations in amplitude corresponded to the failure point it was necessary to use the photographic record as a cross check, which was possible as the framing times of the camera could be referenced back to the timing of the stress pulses as measured by the gauges on the bars. A sequence of images from the experiment generally showed clearly a frame where a crack became visible on the surface of the specimen.
Table 1. Tabulated failure forces across a number of repeated experiments from both high and low strain rate configurations.

| Rod 1 (Instron) | Rod 1 (SHPB) | Rod 2 (Instron) | Rod 2 (SHPB) |
|-----------------|--------------|-----------------|--------------|
| N               | N            | N               | N            |
| 4250            | 5800         | 3724            | 5630         |
| 4370            | 6630         | 3817            | 6130         |
| 4760            | 6750         | 3868            | 6170         |
| 5250            | 7340         | 3875            | 6200         |
| 5260            | 7640         | 4760            | 7260         |
| 5278            | 8500         |                 |              |

The time between this frame and the previous one was assumed to be the time over which failure had occurred. The failure stress was determined by looking for decreases in the recorded stress from the gauge traces over the interval determined from the photographs. While this method was more complex than for the low strain rate experiments it did reliably lead to stress values at the point of failure, as tabulated in table 1. These results again imply that material from rod 1 was on average stronger than rod 2, although it should be noted that there is significant sample to sample variation in the data. There does appear to be a slight strain rate dependence to the failure strength of the material.

The effective resolution of the speckle data was lower than for the Instron experiments, and owing to the need to use a framing camera, significantly less data were able to be taken. However the general pattern (for the SHPB strain data, it was not possible to extract shear information at all) was the same as that for the Instron; an evolution of lateral strain followed, by a failure that had a distinct shear nature to it. It would be beneficial for future investigations to use a higher resolution framing camera and adjust the lighting so as to be able to compare the data at different strain rates more meaningfully. A further improvement to the technique could be to alter the curved anvils that were used. In this investigation the anvils and the sample were sandwiched between the input and output bars of the SHPB system rather than being an integral part of the bars. The coupling between the bar and the curved endcaps is perfect and this could have the effect of increasing the level of the oscillations seen in the voltage traces.

In order to investigate the origin of the shear failure seen in the experimental investigation a modelling effort was undertaken at Georgia Tech. A representative microstructure was imported into CTH 10 [9] in order to attempt to replicate any microstructural effects in the experiments. As the available scan of the microstructure was smaller than the full experimental scale the simulated striker velocity, also using curved anvils was lower than in the experiment to compensate. Overall the simulation had an initial strain rate of 800 with the aim of being equivalent to the Hopkinson bar experiments. While it is obvious that the simulation cannot replicate the larger experimental set-up exactly, the intention was that the early stage failure behaviour might shed some light on the mechanisms of that failure.

The results of the simulation, illustrated in figure 4 show that the failure is dictated by the microstructure of the material, as one might expect. The nature of the failure in the simulation is through the different elements of the microstructure moving relative to each other owing to them being made of different materials. As the elements move small voids are nucleated between them. In the simulation these voids do not form parallel to the loading axis, but in general at an angle to this axis as is seen in the experiment. Unlike the experiment there is not one single dominant crack, however this is likely to be due to the reduced size of the simulation. In the
Fracture growth at 36.6 µs

Figure 4. An image from the simulation showing a number of fractures in the sample. Voids are coloured red in the simulation, and it can be seen that a number of fractures have nucleated and will likely coalesce to form a complete failure of the sample. The fractures are caused by inhomogeneous deformation of the microstructural elements within the simulation.

Figure 5. Electron micrograph showing a network of small cracks in an experimental sample after failure. The lighter of the two material phases is aluminium, the darker nickel. Distinct similarities can be seen with the simulation data, reinforcing the view that this is the correct failure mechanism.

experiment it is likely that a number of these smaller voids coalesce to form the macroscopic crack that was indicative of the failure of the samples. A micrograph of a section of a fractured sample can be seen in figure 5 and shows a network of small cracks, similar to those seen in the simulation. The microstructure does not have a preferential direction within the plane that the images of the experiment and simulation show, though it should be noted that this may not be the case perpendicularly. In fact, the cold spray technique builds up layers and the deformation of the sprayer particles on impact with the existing material is likely to be direction dependent. While it would have been of interest to examine the possibility of anisotropy in these materials, there were insufficient samples available to allow this, and it is something that a future investigation would examine in detail.

4. Conclusions
A number of Brazilian disc tests have been conducted on a cold spray material made of nickel and aluminium. The materials showed minimal strain rate dependence and a tendency to fail in shear rather than in tension as normally expected. The cause of this shear failure is attributed to the microstructure of the material, supported by conducting simulations in the CTH hydrocode. The formation of multiple cracks in the material that are not part of the main (coalesced) failure is predicted by the hydrocode has been verified by microscopy on the failed experimental specimens.

It is clear that when utilising materials manufactured using processes such as cold spraying, care has to be taken with the variations that can occur in the failure strengths of these materials. Possible anisotropy in the materials is should be investigated in more detail, both at the experimental characterisation level, but also at the manufacturing stage. It may be possible to improve the mechanical properties by tuning the fabrication process, particularly given the new insights into the failure mechanisms.
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