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## On the dynamic tensile strength of Zirconium

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**Abstract.** Despite its fundamental nature, the process of dynamic tensile failure (spall) is poorly understood. Spall initiation via cracks, voids, etc, before subsequent coalesce, is known to be highly microstructure-dependant. In particular, the availability of slip planes and other methods of plastic deformation controls the onset (or lack thereof) of spall. While studies have been undertaken into the spall response of BCC and FCC materials, less attention has paid to the spall response of highly anisotropic HCP materials. Here the dynamic behaviour of zirconium is investigated via plate-impact experiments, with the aim of building on an on-going in-house body of work investigating these highly complex materials. In particular, in this paper the effect of impact stress on spall in a commercially sourced Zr rod is considered, with apparent strain-rate softening highlighted.

### 1. Introduction

Understanding of dynamic tensile failure (spall) is of paramount importance for materials employed in environments potentially subject to high rates of deformation (strain). A substantial body of work exists interrogating spall response in both BCC [1] and FCC materials [2]. However, there is a relative paucity of work in the literature looking at spall in HCP materials, where the inherent importance of crystallographic orientation/texture makes such behaviour simultaneously both more difficult to interpret and more important to understand. The majority of such information is focused on the various alloys of Ti – primarily Ti 6(Al) 4(V), which is extensively employed in both the aerospace and defence industries [3]. More recently a resurgence of interest has led to on-going studies into the high-rate response of various Mg alloys [4], which have potential armour applications.

Despite the paucity of available information concerning dynamic failure in Zr, the effects of impurities on its high strain-rate response are relatively well known; even when not directly related to spall, this is of importance as accumulation of impurities at grain boundaries can provide an additional driving force for spall initiation in non-pure metals [5]. Cerreta et al. [6] conducted an extensive study



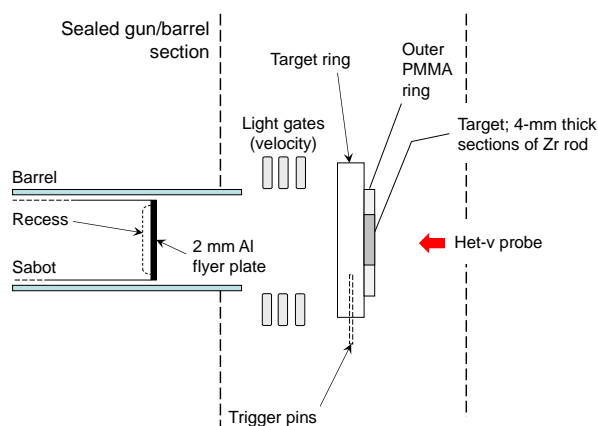
into the effects of oxygen content on Zr and showed that the  $\alpha$ - $\omega$  phase transition increased from 7.1 to 8.3 GPa as the O content increased from <50 to 350 ppm; with the transition entirely suppressed for an oxygen content of 14,000 ppm. This response was attributed to the O atoms, with atomic radii of 0.65 Å, being located at octahedral sites within the tetragonally arranged  $\alpha$ -Zr. As these two locations are able to accommodate atoms of radii 0.66 and 0.35 Å respectively, it was postulated that these large interstitial O atoms displaced space required for the  $\alpha$ - $\omega$  phase transformation within the HCP  $\alpha$ -Zr.

Gray III et al. [7] conducted one of the most comprehensive studies into the dynamic tensile (spall) failure of Zr. Spall was monitored in a cold rolled and annealed high purity Zr plate (54 and 40 ppm of Hf and O respectively) with a strong basal texture and a grain size of c.a. 25  $\mu\text{m}$ . Samples cut both from the plane and through the thickness of the plate were impacted using Cu flyers at 330 m/s (giving a  $\sigma_x$  of 5 GPa). A more ramped initial response and lower HEL were apparent in the through-thickness direction, with the initial nature and magnitude of the spall pullback signal relatively unaffected. Interestingly, however, the re-loading behaviour following the spall pullback minimum had a substantial shoulder/step-like feature in the through-thickness direction which was not apparent in the plane of the plate. This tied in with previous observations of such shoulder-like features (albeit in aluminium) by Chen et al. [8] and was taken to suggest a change in failure mode from ductile-brittle in the through-thickness direction. This conclusion was backed by optical micrographs of recovered material. In both orientations ductile voids were apparent. However, in the transverse case these voids had begun to coalesce with micro-cracking apparent not visible in the in-plane case. This difference was attributed to the Zr behaving in a ‘softer’ manner orthogonal to the transverse direction – with the basal planes preferentially orientated for cleavage.

Notwithstanding the study in reference [7], there is a lack of information available in the literature on the dynamic tensile failure of Zr. Consequently, to enhance this body of knowledge, here initial results from a study into the strain-rate dependence of spall in a commercial Zr rod are presented.

## 2. Experimental approach

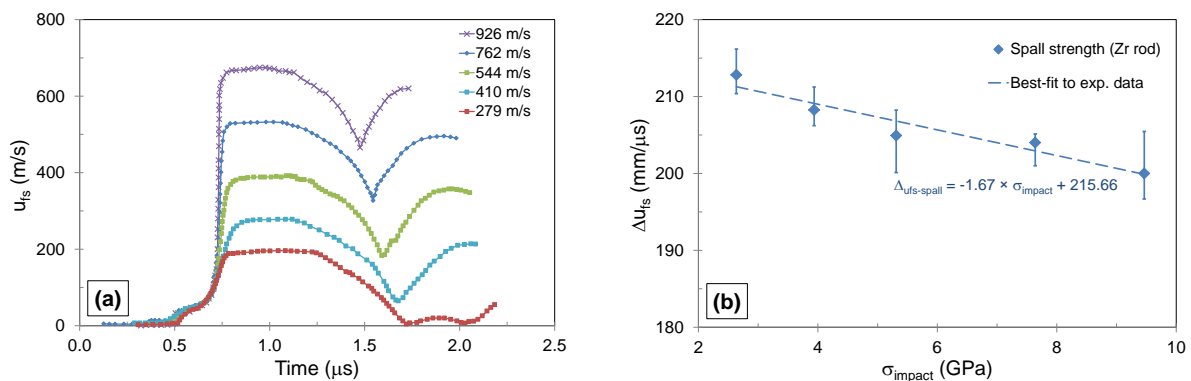
A 5-m barrel, 50-mm bore single-stage gas-gun was employed to accelerate flyer plates at planar Zr targets (from Goodfellow Cambridge Ltd.). These consisted of c.a. 4-mm thick sections of a fully recrystallised – see figure 3(a) –  $\varnothing$ .19-mm commercially pure Zr rod of composition in ppm of 2500 Hf; 1000 O; 250 C; 200 Cr; 200 Fe; 100 N; 10 H. All surfaces perpendicular to the impact axis were finished to a surface roughness of <10  $\mu\text{m}$  ensuring inertial confinement and one-dimensional (strain) loading. Misalignment was confined to  $\leq 1.25$  mrad [9]. Due to their small diameter, the sections of Zr rod were encapsulated within a surrounding PMMA ring using a thin layer of epoxy resin. This approach, previously adopted elsewhere [3, 4, 10], ensured minimisation of flexure on flyer impact; although it is acknowledged that the lack of tensile capture plates etc. will have led to some evolution of microstructural features post-shock. Shock propagation was monitored via a single Heterodyne velocimetry (Het-v) channel [3, 4, 10], with the experimental setup shown in figure 1.



**Figure 1.** Schematic diagram showing the plate-impact experimental setup adopted here.

### 3. Results and discussion

A total of five plate-impact shots were undertaken, each employing 2-mm thick Al 6061 flyer plates. This flyer thickness was chosen to ensure spall plane formation within the target body. In all cases spall planes resulted c.a. 1-mm from the rear target face, with numerical simulation confirming that their formation was uninfluenced by edge reflections. Resultant free surface velocity traces are presented in figure 2(a), with measured pull-back free surface velocities from this data plotted against impact stresses (calculated via the impedance matching technique based on the measured impact velocities) in figure 2(b). The pull-back velocity for the 279 m/s shot was calculated as the sum of the stress differential from the Hugoniot plateau through zero free surface velocity plus the size of the subsequent small ‘peak’. This peak was a function of the inability of Het-v to determine direction of travel (e.g. negative particle velocities).



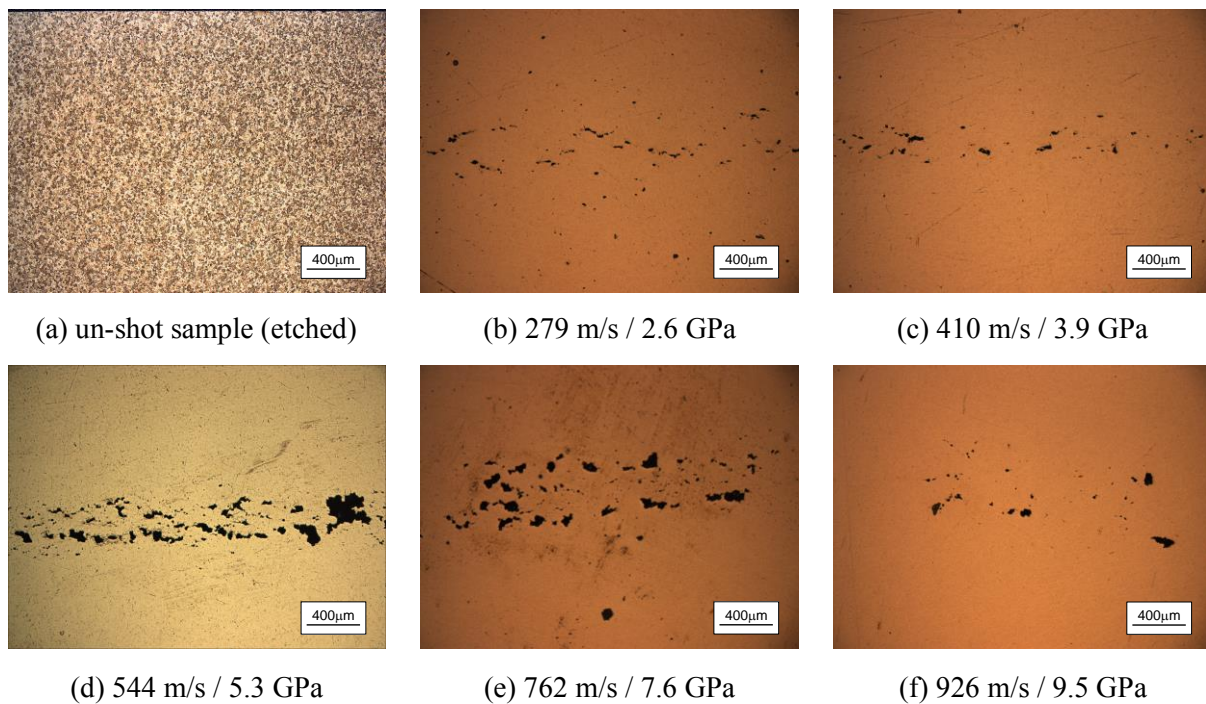
**Figure 2.** (a) Het-v traces showing free-surface velocity profiles following impact of 2-mm thick Al flyers onto 4-mm thick Zr rod targets at a variety of impact velocities; (b) Measured free-surface pull back velocities plotted against calculated impact stress.

Similar to the traces recorded by Gray III et al. [7], in figure 2(a) the HEL is clearly discernible on the rise, before a slightly rounded ramp to the Hugoniot plateau and subsequent release back to the spall pull-back minimum. The HEL appears to be relatively independent of strain-rate at  $38.4 \pm 1.3$  mm/ $\mu$ s and applying the well-known relation  $\sigma_{\text{HEL}} = \frac{1}{2} \times \rho_0 \times c_L \times u_{fs-\text{HEL}}$  can be converted to a stress [6]. Employing Zr material properties presented by Marsh [11] ( $\rho_0 = 6.506$  g/cc,  $c_L = 4.77$  mm/ $\mu$ s), the HEL is calculated as  $0.60 \pm 0.02$  GPa. This is directly comparable to the in-plane value for the Zr plate considered by Gray III et al. [7] which can be calculated via a similar approach as being 0.62 GPa; whereas in the transverse direction the HEL was 1.24 GPa.

The calculated pull-back velocity magnitudes presented in figure 2(b) appear to illustrate a linear decrease in pull-back magnitude with increasing impact stress. Such strain-rate softening has not been explicitly highlighted for Zr elsewhere, although it is consistent with behaviour observed in other HCP materials such as Ti64 [12]. Further, this response is in good agreement with results from a recent study in which the tensile deformation of Zr during Taylor-like impacts was investigated and in which the Zr was shown to exhibit an increase in ductility with impact velocity [13].

The traces shown in figure 2(a) also exhibit an interesting feature during the re-loading following the spall pull-back minimum. This re-loading is relatively gentle/continuous in nature up to 544 m/s, but a ‘step’ or ‘shoulder’ appears to emerge around 544 m/s, becoming marked in the 762 m/s shot, before subsequently reducing in magnitude on the highest velocity 926 m/s shot. Such a change in the post-spall minimum re-loading signal has been previously attributed to a change in failure mode, with a ‘shoulder’ corresponding to a ductile-brittle transition [3, 8]. Analysis of this re-loading behaviour following the spall minimum was undertaken to try and quantify this response. The difference in the re-loading gradient immediately following the spall pull-back minimum and that towards the top of the

subsequently re-loading signal was measured. This was found to be relatively constant in the range  $0.14 - 0.24 \text{ mm}/\mu\text{s}^2$  for impact velocities up to 544 m/s. However, at 762 m/s the marked shoulder-like feature apparent in figure 2(a) led to a significant alteration in this gradient change, to c.a.  $-0.72 \text{ mm}/\mu\text{s}^2$ . At this impact velocity the re-loading gradient was initially very steep up to the apparent shoulder, before becoming markedly shallower. These results suggest that this measure of ‘change in gradient’ behind the spall minimum might therefore be a useful approach to quantify the previously discussed apparent movement from ductile to brittle-like spall failure in this region. Further, in line with the observation that the ‘shoulder’ apparent in the 762 m/s data reduced in magnitude in the post spall minimum response at 926 m/s, the change in gradient in this region returned to a higher value of  $-0.39 \text{ mm}/\mu\text{s}^2$  at this elevated impact velocity. This was taken as tentative evidence of a change back towards a more ductile-like failure mode at this elevated impact velocity. In order to investigate these failure modes in more detail a series of optical micrographs of sections through the spalled samples were taken; these are presented in figure 3 and represent the regions of greatest damage at/about the centre of the samples.



**Figure 3.** Optical micrographs of sections through un-shocked and shocked Zr rod targets.

Figure 3 clearly shows an increase in the degree of damage up to an impact velocity of 762 m/s, where ductile voids are observed to coalesce with increasing impact stress and inter-connected cracks are apparent by 544 m/s (5.4 GPa). However, the fracture morphology appears to subtly change around 762 m/s, with slightly larger and more isolated pores apparent. This ties in with the perceived change in fracture response highlighted in the discussion of figure 2. Further, in figure 3(f) a marked reduction in pore density is apparent. This result – checked via a repeat test (not presented here) – seems to suggest a significant change in spall response compared to the preceding micrographs; although it should be noted that even this degree of damage was lay slightly off-centre – at the very centre of the spall plane virtually no damage was observed. In-line with the previously discussed manner in which the gradient behind the spall minimum changes following the 762 m/s shot, it appears that the failure mode has again become more ductile in nature. While such a change in response has not, to the authors’ knowledge, been observed previously in Zr, it is consistent with the previously discussed work by Cerreta et al. [6] which suggested that the  $\alpha$ - $\omega$  phase change occurs

around 7.1 to 8.3 GPa for grades of Zr with <50 and 350 ppm of O respectively. Given the higher O content of 1,000 ppm here, a phase transition occurring around 9.5 GPa seems potentially reasonable and is one possible explanation for the change in response at the highest impact velocity considered here. However, further analysis of the shocked material will be required to confirm this hypothesis – in particular, account has yet to be taken of the influence of other failure modes such as twinning or shear banding which might be activated in this region, potentially suppressing spall.

#### 4. Conclusions

A total of five plate-impact shots have been undertaken to investigate spall in a commercially sourced Zr rod. The results presented here have provided an additional insight into dynamic tensile failure in this complex HCP material. However, further experiments – in particular analysis of recovered material – will be required before underlying mechanisms can be effectively identified.

The material tested here appears to undergo a significant degree of strain-rate softening – although at this stage the authors' have yet to propose a mechanism to account of this response. In addition, analysis of the post-spall minimum behaviour in the recorded free surface velocity traces suggested that such data may provide evidence of changes in spall failure mode – and potentially in material phase. This analysis qualitatively agreed with the interpretation of the optical micrographs which appeared to show an increase in spall (pore formation and coalescence) up to around 7.6 GPa, with a change in failure mode at elevated stresses. Further, it was noted that this change in behaviour appeared to be occurring at a stress consistent with the observed emergence of  $\alpha$ - $\omega$  phase change elsewhere in the literature.

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