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Steam Performance of $\text{UB}_2/\text{U}_3\text{Si}_2$ Composite Fuel Pellets, Compared to U_3Si_2 Reference Behaviour

Turner, J, Abram, T.J.

Nuclear Fuel Centre of Excellence, The University of Manchester, United Kingdom

Abstract

Uranium diboride (UB_2) was produced via arc melting and mixed with U_3Si_2 material via cold-press and sintering. Composite materials, from 0-100% UB_2 were placed in high temperature flowing steam and mass gain observed. It was seen that the addition of 10 wt% UB_2 to U_3Si_2 increased the onset of reaction by 100°C compared to pure U_3Si_2 in the same conditions. 50 wt% $\text{UB}_2/\text{U}_3\text{Si}_2$ pellets maintained this increase, alongside a significant reduction in the ejection of material typically seen in U_3Si_2 -steam reactions. From these results, a mechanism for both composite reaction and the performance of U_3Si_2 in steam is proposed.

Keywords: Nuclear Fuel, Accident Tolerant Fuel, Uranium silicide, Uranium boride

1. Introduction

Accident tolerant fuels (ATFs) are the current subject of a wide ranging international research effort. ATFs include a range of alternative claddings and high-density fuel materials, with the aim to reduce stored energy in the fuel, reduce enrichment requirements, and eliminate the potential for hydrogen production during severe accidents. Uranium silicide fuel, specifically the U_3Si_2 phase, is of interest as an ATF material due to a high thermal conductivity, uranium density and compatibility with ceramic cladding concepts [1]. However, recent results suggest that U_3Si_2 undergoes a rapid and energetic reaction with high temperature steam [2]. This reaction is so energetic that measuring mass gain is problematic due to material ejection during the experiment.

As an alternative, uranium diboride (UB_2) has several advantages; a high uranium density (11.7 g/cm^3) [3], good predicted thermal conductivity [4] and greater resistance to high temperature water/steam than both U_3Si_2 or uranium nitride fuel materials [5]. Historically UB_2 has received limited interest, as boron is a strong thermal neutron absorber. However, the commercial production of enriched boron-10 for use in control rods necessarily produces material enriched in boron-11 as a by-product, which has a very small neutron cross section. There is currently renewed interest in UB_2 fuel concepts, most notably though atomic scale modelling to predict physical properties [4, 6].

The creation of a composite U_3Si_2 and UB_2 material is of interest for two reasons; it offers the potential to improve overall water performance of U_3Si_2 while maintaining a high uranium density, and the provision of a distributed burnable poison within U_3Si_2 which does not disrupt the uranium bearing phase (achieved by varying the boron-10 to boron-11 ratio). Burnable poisons in U_3Si_2 may be problematic with alternative solutions explored to date proving unstable or incompatible with U_3Si_2 [7]. Maintaining some silicide component within the fuel may be of

interest to reduce the economic penalty that arises from the need to use enriched boron within UB_2 .

The performance of a composite UB_2/U_3Si_2 material under high temperature steam has been investigated to determine if it offers any improvements compared to reference U_3Si_2 and to develop a greater understanding of the U_3Si_2 -steam reaction.

1.1. Methodology

Composite discs were produced based on the general manufacturing route for U_3Si_2 pellets [8]. A small amount ($<5\%$) of a $UO_{0.75}C_{0.25}$ phase was distributed throughout the composite pellets, which formed as a result of a small fraction of uranium metal present in the arc melted UB_2 . This phase was observed in UB_2 following high energy milling in a tungsten carbide milling vessel and was assumed to have formed as a result of uranium metal interacting with the carbide walls. It did not appear to increase in mass fraction following production of composite pellets. Carbide phases are known to be highly reactive with water even at low temperatures and so the material containing this phase was considered to form a lower bound of steam performance. Material successfully produced without carbide inclusions could be expected to show improved steam performance at similar temperatures. The manufacture of these composite materials is detailed more completely in [9].

For steam testing, fragments were mechanically broken from the sample and placed in a cylindrical alumina crucible (approx 6mm x 6mm). Thermogravimetry (TGA) was performed using a Netzsch 449-STA 'Jupiter' F1. Samples were brought to 250 °C in flowing argon and held for 10 minutes before introducing flowing steam at a rate of 5g/hr while maintaining argon flow. The furnace temperature was increased to 1000 °C at a rate of 20 °C/min. Furnace temperature was controlled to within 5 °C, while sample temperature was monitored and is reported here.

Previous work on the silicide-steam reaction has highlighted the importance of crucible size and shape [2]. It was suggested by Sooby Wood that deep crucibles hinder the reaction via the production of a localised atmosphere around the sample. This was examined via the use of zirconium sponge as a test sample, which was seen to undergo a reduced reaction within the standard deep crucible supplied.

A similar test was performed on zirconium sponge samples within the present work and compared to the modified crucible employed by Sooby Wood (Figure 1). The earlier onset temperature and greater overall mass gain observed in the present work suggest that the crucible employed does not inhibit the steam reaction.

The straight walled crucible used here was preferred in an effort to limit mass loss events which were expected when exposing U_3Si_2 to steam at these temperatures. The fragments tested were relatively small and placed at the bottom of the crucible, thus limiting the ejection angles which could remove sample mass from the crucible entirely.

Samples were examined via X-ray Diffraction (XRD, Panalytical Empyrian) and Scanning Electron Microscopy (SEM, FEI Nova NanoSEM). XRD was performed on powder produced via crushing samples in an agate pestle and mortar.

For clarity within the present work we describe samples which have been exposed to steam as 'oxidised', as the end product from this reaction is UO_2 . This is not intended to imply that the only reaction occurring is that of oxidation.

Figure 1: Test exposure of zirconium sponge to steam within the crucibles employed from the present work, the rapid reaction suggests that the crucible geometry employed does not hinder the reaction noticeably. Included for comparison is the data on zirconium sponge oxidation in a modified crucible, reproduced from Sooby Wood et al [2]

Material	Onset Temperature (°C)
U_3Si_2	453, 449*
UB_2	629
50wt% UB_2 Composite	548, 575
10wt% UB_2 Composite	553*

Table 1: Temperatures at which rapid mass changes were observed for materials listed. * denotes a determination made manually and not using the automated software routine

2. Results

2.1. Thermogravimetry

Figure 2 shows the change in mass for samples exposed to flowing steam between 250 °C and 900 °C. For reference, data from [2] is also shown. In line with existing literature on the silicide-steam reaction, percent mass changes are reported here rather than normalising reaction rates to surface area, due to material loss which was observed on some measurements. Spikes visible on the data are due to water condensing at the outlet of the furnace, temporarily preventing flow. Although these have been numerically smoothed using the Netzsch Proteus software, small sharp peaks on the plots remained. Where possible onset temperatures were calculated from this analysis software and are presented in Table 1. In some cases (denoted * in Table 1) peaks caused by condensation prevented this, and a best estimate was made manually.

Onset of the silicide reaction appears to be in good agreement with the literature U_3Si_2 reaction, although the response is different as limited material ejection was observed within the present work. Data in the magnified inset of Figure 2 shows a slight increase in mass of U_3Si_2 samples examined as a function of the present work, while the literature data begins to lose mass, likely as a result of small mass ejection events. The slightly deeper crucible employed within this

Figure 2: Thermogravimetry of composite and reference fuel materials in flowing steam

work appears to have limited the outright loss of material. A mass loss event is visible on one U_3Si_2 reference sample, although at a higher temperature than previously published data. We consider this data to be in good agreement with published data given that the crucibles are more likely to retain fragments due to their vertical walls, and the mass uptake observed in Figure 2 correlates well with the temperature of ejection previously reported.

50wt% UB_2 content has a significant increase in onset temperature compared to the reference silicide material. Very slow mass gain is observed between 500 and 550 °C for these samples, before the onset of a more complicated multi-stage mass gain reaction, culminating in mass loss above 750 °C.

This is similar to the behaviour observed in the reference UB_2 sample; a three stage reaction, with an inflection point around 725 °C and mass loss above 750 °C. The composite fuel begins to react at a lower temperature than UB_2 , but significantly above the U_3Si_2 onset temperature. It's behaviour varies from that which would be expected if both constituents were reacting separately, and very limited mass gain is observed until above the temperature at which the pure U_3Si_2 reaction with steam has plateaued. The variation in sample behaviour visible between the two 50wt% composites is likely due to differences in surface area to volume ratio, as the fragments varied in shape and size.

2.2. X-ray Diffraction

All XRD patterns from material following exposure to 900 °C steam show only the production of UO_2 . No compounds of boron or silicon were observed (Figure 3) aside from a very small quantity of UB_2 which remained within the 50-50 composite material (highlighted as '*' on Figure 3). This suggests that either any silicon and boron compounds that formed were volatilised and escaped the sample, or that amorphous compounds were formed that cannot be detected by XRD.

Figure 3: XRD analysis of materials following exposure to high temperature steam. All peaks aside from those denoted * represent UO_2 . Peaks marked with * indicate very small amounts of remaining UB_2

2.3. Microscopy

Electron microscopy consisted of three sample sets; material which had not been exposed to steam, material briefly exposed to steam at one of two fixed temperatures, and powdered material produced from exposure to 900 °C steam.

2.3.1. Non-Exposed Materials

Figure 4 shows a 50wt% composite (A), sintered UB_2 microstructure (B) and reference U_3Si_2 (C) which have not been exposed to steam.

Figure 4 C shows a region representative of the bulk sintered U_3Si_2 material produced for this study. One region of the produced pellet showed significant porosity along the edge of the pellet (Figure 5) which may result from the pellet being placed in contact with an oxygen getter (tantalum foil) during sintering. The presence of porosity in steam-corroded U_3Si_2 was therefore not treated as conclusive evidence of reaction, despite the apparent similarity between pores on Figure 5 and evidence of corrosion presented in [2].

In line with the expected sintering performance of the U_3Si_2 powder employed, which was several weeks old at the time of sample production, a number of oxide regions are also visible within the non-exposed material. These were identified as localised grains of UO_2 , and proved relatively easy to distinguish from oxide produced from interaction with steam as a result of their morphology. The oxide phase present in the non-exposed samples formed small circular regions, typically around 5 μm in diameter.

Figure 4 A shows the microstructure of a 50wt% composite pellet. Regions of U_3Si_2 (lighter) and UB_2 (darker, rectangular) are distinguishable, as are small grains of UCO phase discussed earlier. The density of the composite material was similar to the reference U_3Si_2 material, and appears to be an artefact of the age of the silicide powder used to produce the composites. This is thought to result from the formation of an oxide layer during storage, even in the sub-ppm

Figure 4: A - Microstructure of 50wt% composite material. B - UB_2 . C - U_3Si_2 . All produced materials show some secondary phase impurities porosity, generally resulting from the age of the silicide powder employed or the presence of UCO

O_2 environment of the inert argon gloveboxes employed here. There is no detectable interaction layer between the boride and silicide phases following sintering.

Figure 4 B is representative of the sintered UB_2 microstructure, which contains grains of UB_2 and inter-granular UCO regions. The microstructure in Figure 4 C was produced after sintering for 1 hour at 1800 °C due to the relatively high melting point of UB_2 , as opposed to the silicide and composite materials which were sintered at 1400 °C. It is likely that higher density could be achieved with longer hold times and/or higher sintering temperatures, but this was beyond the scope of the present study.

2.3.2. Short-Exposure Materials

In order to understand the change in steam-response observed during TGA, fragments of U_3Si_2 and 50wt% composite material were exposed to steam for five minutes at fixed temperatures, below that at which they were expected to pulverise. U_3Si_2 was exposed at 465 °C and composite material was exposed at 465 °C and 565 °C.

Following five minute exposures at 465 °C, there appears to be a clear difference in silicon behaviour between U_3Si_2 and composite material. Figure 6 shows the exposed surface of a U_3Si_2 fragment with both SEM and EDX maps for the location of silicon, uranium and oxygen. The top right of the images within Figure 6 show material which has broken away from the

Figure 5: Band of porosity visible adjacent to surface of U_3Si_2 .

bulk surface, which appears to have fractured behind the oxide layer, exposing more silicide-containing material. Within the bulk of the fragment, silicon rich regions are forming at a sharp edge to the existing oxide layer. These silicon rich regions are similar in size and morphology to cracks which appear to develop at later stages of the reaction, as seen on Figure 8, which also shows striations typical of hydride formation, and an area of UO_2 containing a very large number of cracks. Each of the individual silicide pieces visible in Figure 8 is surrounded by oxide, which appears to progress from the cracks inwards, further displacing Si. Similar morphology is visible on parts of the fractured material in Figure 6, which culminates in a further silicon-rich small central region, completely surrounded by stable oxide.

In contrast, Figure 7 shows an open crack on the surface of a 50wt% composite fragment. Here silicon rich regions appear to form in bands across the U_3Si_2 grain towards nearby UB_2 grains. The existing crack has a layer of UO_2 directly adjacent to it, similar to those which have formed adjacent to cracks on U_3Si_2 shown on Figure 8. No silicon rich regions were observed forming at grain boundaries, although they do appear to form at the centre of heavily oxidised grains.

Following exposure to 565 °C steam, small solid fragments of 50wt% composite remained, although a large proportion of the original sample had formed UO_2 powder. Representative images from the edge of these solid fragments are shown in Figure 9 and Figure 10. At this temperature, which is within the rapid mass gain phase seen in Figure 2 for these samples, silicide grains within the bulk can be seen to have begun to oxidise. In contrast to the slow inward growth of oxide seen at lower temperatures, in Figure 9 UO_2 appears to form as striations across the silicide material, possibly due to the distortions caused by the formation of the $U_3Si_2H_2$ phase. UB_2 grains can be seen to have broken away from the bulk of the sample, and but show limited oxidative attack at this temperature.

It can be seen in Figure 10 that layers of material have been removed from the sample, but appear to have maintained cohesion and form a solid single layer. EDX suggests that oxygen has begun to attack silicide within the layers, but not caused the breakaway of small individual

Figure 6: SEM and EDX of U_3Si_2 after short exposure to steam at 465 °C

Figure 7: SEM and EDX of $\text{UB}_2/\text{U}_3\text{Si}_2$ composite after short exposure to steam at 465 °C

Figure 8: SEM image of U_3Si_2 after short exposure to steam at 465 °C, this region appears to have corroded more than that shown in Figure 6 and cracks are visible at several points, exposing all sides of the silicide grains.

Figure 9: SEM and EDX image of 50wt% composite fragment after exposure to 565 °C steam for five minutes.

Figure 10: Low magnification SEM and EDX image of 50wt% composite fragment after exposure to 565 °C steam for five minutes. The high oxygen content between the uranium bearing layers is a result of the resin used to mount the sample.

grains seen in on U_3Si_2 .

2.3.3. Powder Morphology Following 900 °C Steam

In all cases following exposure to 900 °C steam, samples were reduced to powder which was confirmed to be UO_2 via XRD. SEM micrographs of the final powder products are shown in Figure 11.

Figure 11 A shows UO_2 formed by reacting U_3Si_2 with steam. In contrast to the UB_2 -steam reaction (Figure 11 B), it shows a large number of branching cracks on all surfaces, creating a large surface area. Figure 11 C is the result of reacting a 50wt% composite with steam, and shows a limited number of branching cracks, although some are present on exposed surfaces. Figure 11 D shows a backscattered image of a 50wt% composite following steam exposure. The dark regions visible within the grain were identified via EDX as a boron-silicon mixture, which was observed on several similar powder grains, often visible towards the centre of crystallites. Visually it appears that at later stages of the reaction, UO_2 forms a semi-stable surface around a 'core' of lighter elements, which do not react further. It is possible therefore that light elements are not seen on XRD after steam exposure due to limited penetration of X-rays into uranium-bearing phases. Estimated penetration of a typical XRD X-ray to UO_2 is around $6\mu\text{m}$, and would not reach the centre of the features seen on Figure 11 D.

3. Discussion

The addition of a significant mass of UB_2 to U_3Si_2 greatly increases the temperature at which it begins to rapidly react. From TGA, this effect is beyond that which would be expected if the material was partially reacting at lower temperatures, for example due to the lower mass fraction of U_3Si_2 present within the material. Indeed, there is no measurable increase in mass at the temperatures at which complete pulverisation of U_3Si_2 is observed.

The mechanism by which this improvement is achieved requires further understanding of the U_3Si_2 -steam reaction; it cannot be explained if the formation of $\text{U}_3\text{Si}_2\text{H}_2$ is the sole reason for the early onset and energetic nature of the reaction, as has been theorised by Sooby Wood amongst others [2].

Figure 11: SEM Micrographs of powder produced from following steam tests of; A - U_3Si_2 , B - UB_2 , C - 50wt% composite, D - 50wt% composite

As previously discussed in the literature, this is a complex reaction which is known to at least partially result from the formation of a $U_3Si_2H_2$ phase, that is theorised to lead to rapid oxidation due to the distortion of the U_3Si_2 lattice. Within the present work we have observed similar striations on silicide material exposed to high temperature steam, both in composite and reference U_3Si_2 material, and so consider the present work to show good agreement with literature in this respect.

However, microscopy of U_3Si_2 exposed to steam at 465 °C shows the formation of silicon rich regions which closely resemble the morphology of cracks observed at high temperatures, and appear to follow grain boundaries. From the separated silicide/oxide grains visible on the same observations, we suggest that these may form failure points which expose large regions of unreacted silicide material for further reactions. The presence of large amounts of silicide within the material removed from the bulk appears to preclude this relating solely to the spallation of an oxide layer formed on the surface.

One proposed mechanism by which this might occur is through the production of volatile or brittle silicon compounds at grain boundaries. There are numerous Si-O-H compounds which would be volatile at the required temperatures. This would also go some way towards explaining the limited silicon observed in the end products of the steam reaction, and the energy at which material has been seen to eject from apparatus during silicide steam reactions. However, cracks at the silicide surface and removed mixed oxide/silicide material were much more commonly observed than silicon-rich structures within the bulk. Although the morphology of cracks open to the surface is similar to that of the silicon-rich regions which were observed, we acknowledge that further work is certainly required to demonstrate this mechanism, particularly given the current

importance of the steam-silicide reaction for the deployment of accident tolerant fuels. The absence of silicon compounds following oxidation silicide in both air and steam is an ongoing challenge for the understanding of this important mechanism [10, 2, 11].

An alternative mechanism is through the formation of borosilicate glass on the material surface. This would likely be resistant to steam corrosion up until its melting point, typically around 500-600 °C. No such layer was observed via high-magnification SEM and EDX, but this does not preclude the presence of such a feature, instead setting an upper limit on its thickness to be below that detectable via SEM (around 500 nm). Silicon does appear to diffuse toward boride grains within composite pellets which would provide the requisite materials for the formation of a borosilicate layer alongside oxygen.

Given the complex nature of the silicide-steam reaction, it is not surprising that the mechanism by which UB_2 improves upon this appears to be complex and is not yet fully understood. Although the rapid rate of reaction has previously been linked with hydride formation, interrupting this process does not appear to be the mechanism by which the composite limits degradation, as hydride-striations were regularly observed on post-exposure silicide grains within the composite.

The addition of a significant weight percentage of UB_2 to U_3Si_2 does appear to prevent the formation of a large number of cracks within the material as seen at low and high temperature exposures as well as the powder produced following 900 °C exposure. Although it does not prevent their formation altogether, it limits their number significantly, preventing a rapid increase in surface area.

This may be related to the behaviour of silicon during the composite reaction, which was seen to form multiple bands of silicon rich material across silicide grains within the composite material. Grains in which these bands were observed also showed striations which were taken to show the formation of the $U_3Si_2H_2$ phase and it is proposed that silicon displaced by the oxidation reaction at the surface is able to occupy sites within the $U_3Si_2H_2$ lattice more easily than at a U_3Si_2/UB_2 boundary.

At higher temperatures, these bands develop into mixed oxide-silicide grains. It has been previously established that the introduction of hydrogen to the U_3Si_2 distorts the lattice, causing significant swelling and allowing oxygen ingress more easily [12]. We therefore suggest that the mixed-grains observed are a result of oxygen occupying the lowest energy sites first. It is not clear where the displaced silicon from this reaction relocates to, although it has not been detected in either reference material or composite exposed to 900 °C.

We theorise that the diffusion of silicon within the grain, rather than to the grain boundary, acts to increase the amount of surface oxidation (and hence silicon displacement) required to form regions to open into cracks later in the process. Further work is clearly required to understand this mechanism and the underlying silicide-steam reaction in greater detail.

It is also clear that UB_2 grains within the composite are largely unaffected by steam at temperatures at which U_3Si_2 is converted to UO_2 . Although little data exists on this material, predictions of its behaviour and thermal properties are extremely encouraging, and the results presented here suggests that it may offer significant improvements to the resistance of U_3Si_2 to oxidation as a result of fuel-coolant interactions.

4. Conclusions

A number of composite U_3Si_2/UB_2 pellets were produced and their performance tested in high temperature steam. The addition of small amounts (10wt%) of UB_2 to U_3Si_2 increases

the onset temperature of the steam reaction by around 100 °C, and the addition of 50wt% UB₂ maintains this increase while limiting the amount of material ejected from the crucible. Both UB₂ and the composites tested here appear to offer substantial advantages over U₃Si₂ as an accident tolerant fuel for light water reactors.

Boron enrichment is a well established technology, hence the availability of boron depleted in boron-10 offers the prospect of both an acceptable neutronic performance and the means of incorporating an integral burnable absorber within the fuel.

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