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CHARACTERIZATION OF CHARGED PARTICLE BURSTS FROM DEUTERIUM LOADED THIN TITANIUM FOILS

D.H. Beddingfield, F.E. Cecil, C.S. Galovich, H. Liu[a]
and Sally Asher[b]

[a]Colorado School of Mines, Golden CO 80401 USA
[b]Solar Energy Research Institute, Golden CO 80401 USA

Following our recently reported[1] observation of intense bursts of charged particles from deuterium gas load thin Titanium foils, we conducted a relatively exhaustive analysis of the samples involved in this study in order to better understand the gas loading process, to characterize the elemental and structural properties of the samples, and to ascertain, if possible, any differences between those samples which evinced particle bursts and those which did not.

Our samples consisted of Ti662 (6%V, 6%Al, 2%SN)[2] lathe turnings of nominal thickness about 100 μm and of surface dimension about 1 cm. by 2 cm. The D_2 gas was introduced by first annealing the samples at 700° C for three hours under vacuum and then introducing 1 Atm of D_2 and allowing the samples cool slowly. An important dimension of the present work is to document the various techniques employed to measure the deuterium-metal ratio and its depth profile. These techniques included gravimetric analyses, X-ray diffractometry (XRD), and secondary ion mass spectrometry (SIMS).

The gravimetric analyses consisted of careful weighing of the samples before and after the gas loading for both the deuterium samples as well as the hydrogen control samples. The results of these weighings are given in Table 1. The D to metal ratio was found to be generally less than unity for the D_2 samples and typically greater than unity for the H_2 samples. Since identical preparation procedures were followed in both sets of samples, the observed loading increase in the case of the H_2 samples suggest greater mobility of the H_2. The one D_2 sample with D-metal ratio significantly greater than unity (D-IV) consisted of much thinner (about 30 μm)

turnings. This would suggest that the thicker samples were not uniformly loaded with depth and that, in fact, the near surface regions consisted of D-metal ratios somewhat greater than unity.

Table 1. Gravimetric Analyses of H and D Samples

Trial	% mass increase	Gas-metal atom ratio
H II	1.86	0.86
H IV	3.02	1.39
H V	3.08	1.42
D I	3.70	0.85
D II	3.12	0.72
D III	4.70	1.09
D IV	5.57	1.29
D V	2,45	0.57
D VI	2.49	0.58
D VII	2.84	0.66
D VII	2.14	0.49
D IX	3.22	0.74

An expected consequence of the gas loading would be an increase in the inter-atomic spacing in the metal. This was verified by XRD in which the Ti662 beta-phase lattice parameter was compared to the deuteron-metal atomic ratio as determined from the gravimetric analyses described above. This comparison is shown in Figure 1 where a definite positive correlation in noted.

This expected swelling was likewise noted in a series of scanning electron micrographs of the samples. One such micrograph is shown in Figure 2. Not shown are the pre-loading micrographs of the sample which indicated surfaces flat at the sub-micron level, featureless except for the lathe tool markings. The rumpled nature of the surface in Figure 2 is qualitatively consistent with the results of the XRD investigation noted above.

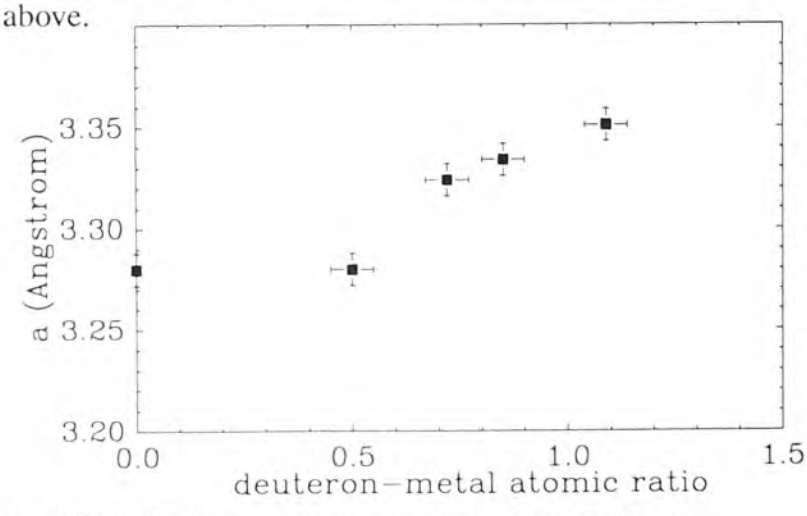

Figure 1. Comparison of Ti662 β lattice parameter versus D-metal ratio.

A semiquantitative elemental analysis of the near surface region of the samples was afforded by the SIMS study. One such depth profile, for one of the D_2 samples, is shown in Figure 3. The maximum sputtering time of 100 minutes corresponds to a depth of about 2 μm. In addition to the Ti, Sn, V, and Al, the presence of the H and D are noted. The low level of H in the D sample is consistent with the nominal abundance of hydrogen in our deuterium gas bottle. Not surprisingly, a surface layer of C appears. Because the primary ion beam in the SIMS facility was O, any surface or near surface oxidation of the sample could not be identified. There was no noticeable difference, again down to the 2 μm level, between the SIMS profiles for those D samples which produced charged particle bursts versus those which did not.

The neutron activation analysis afforded a more detailed and quantitative analysis of the samples although this technique was insensitive to any spatial inhomogeneities. The results of this analysis are given in Table 2. Again the dominance of Ti, Al, V, and Sn are noted. Certain light elements such as H, D, Li, C or O cannot be detected with reactor activation analysis and are consequently not tabulated. In this table the elemental analysis for a typical sample which produced one of the charged particle bursts described in Ref. 1 is compared to a sample which produced no bursts. There is no compelling evidence suggesting that a given element or elements might be responsible for the production of the charged particle bursts.

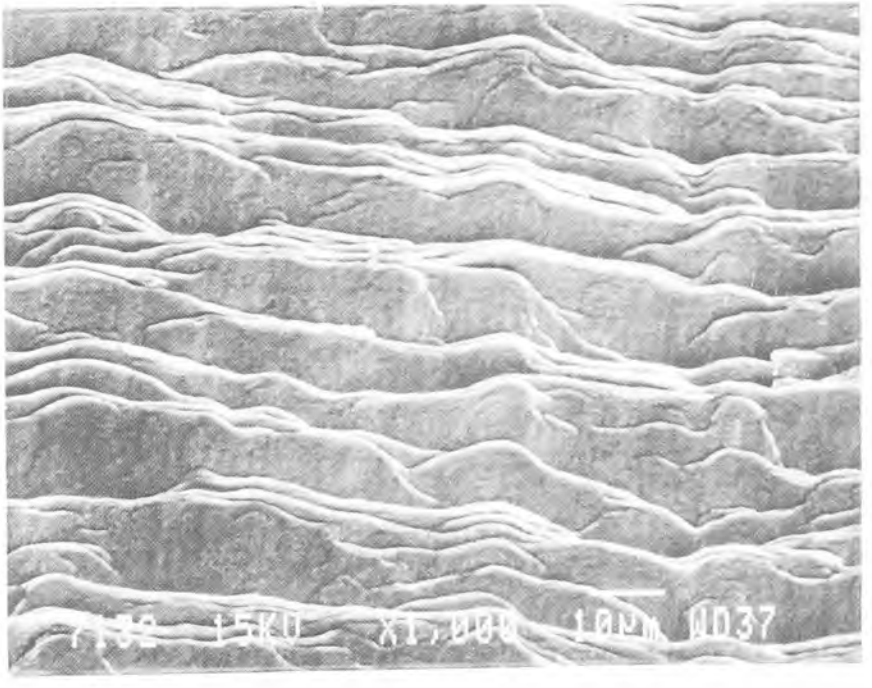

Figure 2. Scanning electron micrograph of D-loaded Ti662 surface.

Table 2. Summary of Neutron Activation Analysis

Element	No burst (ppm)	Burst (ppm)
Ti	760800	713200
Al	68860	58640
V	49700	46230
Sn	15760	15160
Fe	4697	4603
Cu	4611	3624
Dy	136	139
Ni	103	135
Mn	77	127
Cr	81	89
Tm	24	22
Mo	19	21
Na	40	19
As	15	17
Ga	10	11
Sb	7	6
Sc	4	4
Co	3	3

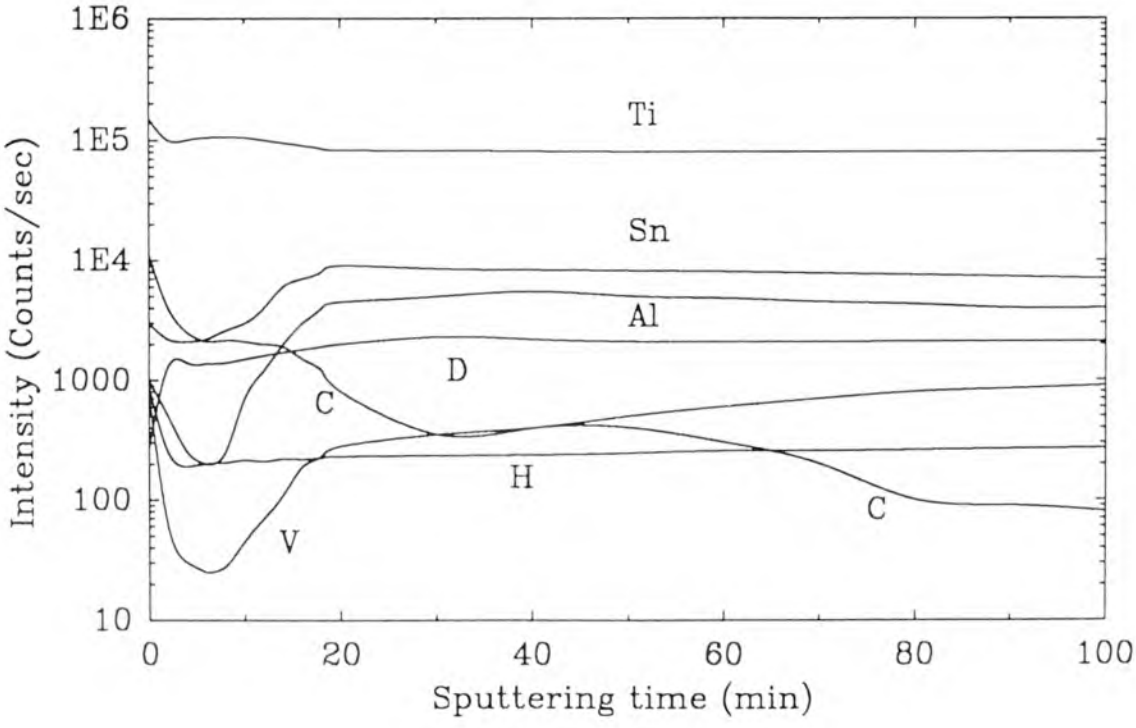

Figure 3. SIMS Elemental depth profile of one of the Ti-D samples.

In conclusion, the studies which we have carried out on the hydrogen and deuterium gas loaded Titanium foils indicate that we employed a reliable and reproducible gas loading technique, capable of achieving gas-metal ratios of order unity to depths of at least several microns and probably more. No differences, however, were noted between those sample from which charged particle bursts were observed versus those which did not.

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References

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