The production of multi-element opacity targets for x-ray laser experiments

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Introduction

Recent experiments on the Vulcan laser system at RAL obtained opacity measurements of hot dense iron plasma using an x-ray laser[1]. In the experiment the plasma was generated from targets containing buried iron layers. A recent follow on TAW Vulcan experiment required the production of thin iron foil targets doped with aluminium (10 atomic-%). The targets were specified to be a thin (50nm thick) metallic layer on a plastic backing and this foil was to be stretched across a small hole to act as a support. Targets had a range of thicknesses of plastic and additionally some targets were 100% iron and some had the 10% aluminium content. Although for previous related experiments iron and aluminium had been coated as separate layers targets had not been made from a co-deposit. It was also requested that the co-deposit was characterised for elemental composition.

Deposition technique

To carry out the co-deposit required the use of the dual-head sputtering system in the Target Fabrication Laboratory. The system contains 2 magnetron sputtering heads; one with a normal strength magnet for the sputtering of non-ferrous materials and the other with a high strength magnetron sputtering head for magnetic materials. The heads can be used with any combination of 3 power supplies (2 × DC and 1 × RF). The design of magnetron heads incorporates a magnetic field of sufficient strength to extend beyond the target material and thereby confine the discharge plasma near the target surface. This designed constraint allows the sputtering process to be carried out at lower pressures than non-magnetron heads and achieves higher sputtering rates. The standard (non-ferrous) 2" magnetron sputtering head was used with the RF power supply as aluminium coatings can easily be produced using this configuration. The high strength magnet head was used to carry out the iron sputtering. The high strength head was required because strongly magnetic materials tend to distort and even eliminate the magnetic field that is required to confine the plasma near the target material[2]. The distortion or elimination of the field means that sputtering cannot be achieved at the lower process gas pressures available in our system. Furthermore the high strength head was used in conjunction with the DC power supply as RF power supplies used with high strength magnets can decrease the deposition rate[3].

To produce a good quality co-deposit it is essential to understand how different materials behave under varied deposition conditions and the subsequent coating rates that can be achieved. The deposition rates of each material were studied independently with the gas pressure kept constant throughout trials for both materials. Results are shown in figures 1 and 2.

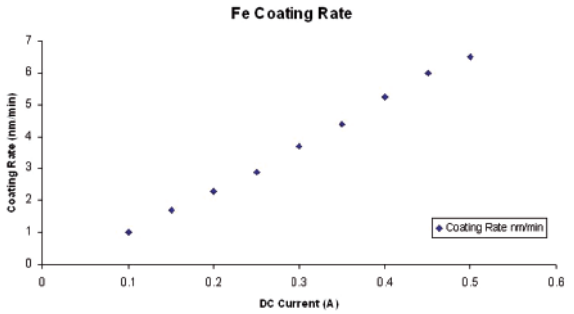

Figure 1. Iron coating rate as function of current.

Figure 1 shows the current dependence of the iron coating rate and illustrates that it is easily possible to achieve rates of up to 7nm/min using the DC power supply at a relatively low current of up to 0.6A. For the ratio of materials required (10% aluminium: 90% iron, by atomic composition) using a modest coating rate of 6.5nm/min for iron it was necessary to be able to coat aluminium at a rate of approximately 0.72nm/min (allowing for density assumptions for the deposited materials). The deposition curve of aluminium using an applied RF power is shown in figure 2.

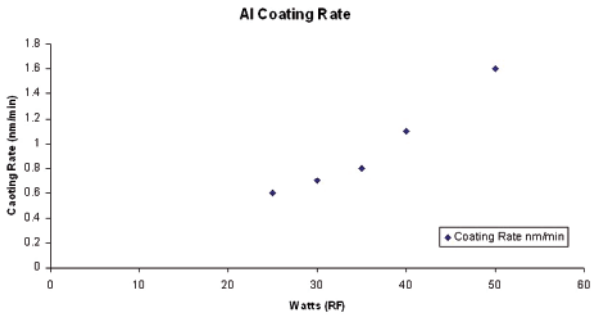

Figure 2. Power dependence of aluminium sputtering rate.

It can be seen that a rate of 0.72 nm/min is achievable with an RF power of 31 Watts.

Deposit characterisation

A trial 50nm thick co-deposit run was carried out as described above and the resultant coating was characterised while still on its glass substrate using a Topcon SM-200 SEM with EDS/EDX (energy dispersive x-ray spectroscopy) capabilities. The sample was analysed for elemental content of aluminium and iron. The spectrum that was produced is shown in figure 3 and the raw data of the percentage composition by weight and atomic percentage of each element is shown in figure 4.

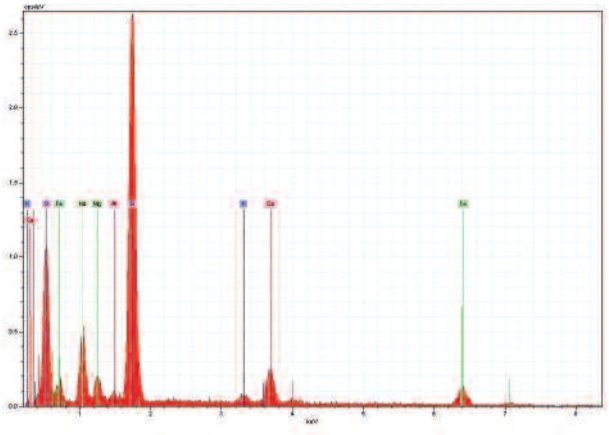

Figure 3. Elemental analysis spectrum from Fe:Al co-deposit.

Spectrum: Acquisition

El	AN	Series	unn. C (wt. %)	norm. C (wt. %)	Atom. C (at. %)	Error (%)
Si	14	K-series	27.10	27.73	20.27	1.2
Na	11	K-series	8.29	8.49	7.58	0.7
Fe	26	K-series	5.41	5.54	2.04	0.7
Ca	20	K-series	3.72	3.80	1.95	0.4
Mg	12	K-series	2.16	2.20	1.86	0.3
K	19	K-series	0.67	0.69	0.36	0.4
Al	13	K-series	0.40	0.41	0.31	0.2
O	8	K-series	49.99	51.14	65.63	9.8
Total:			**97.74**	**100.00**	**100.00**	

Figure 4. Compositional analysis data from Fe:Al co-deposit.

Bearing in mind that in EDS the majority of x-ray excitation originates from a volume beneath the surface (of, say, notionally 1μm diameter) it is to be expected that most of the x-rays detected would be from the underlying glass substrate. The data in figure 4 shows large amounts of silicon and oxygen which presumably originates from the glass substrate. Trace elements and, possibly, contamination, presumably account for the other elements (Na, Ca, Mg, K) detected. A ratio by atomic percentage of iron at 2.04% and aluminium at 0.31% (assuming negligible content of iron or aluminium in the glass) was measured in the co-deposit. This gives a corrected percentage of 13.2% aluminium.

Technique refinement

To produce a co-deposit closer to the requested specification a second coating run was performed and the characterisation data from the deposit is shown in figures 5 and 6. In the second run the deposition rate of the iron was kept the same as in the first run but the RF power used to deposit aluminium was reduced to 25 Watts.

The data was collected for the second coating run from a sample that had been mounted onto a copper target mount. For reasons previously discussed in EDS analysis the copper signal is not surprisingly very dominant in the data but none the less Fe and Al lines can be quantified. A number of sets of data were taken and the data shown in figures 5 and 6 is representative of the average data.

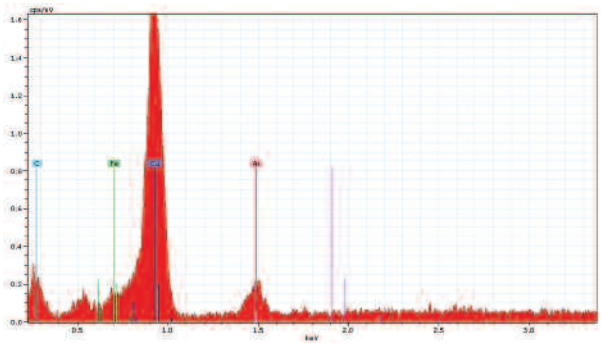

Figure 5. Iron and aluminium spectral data from the second run (sample mounted on copper holder).

Spectrum: Acquisition

El	AN	Series	Net	unn. C (wt. %)	norm. C (wt. %)	Atom. C (at. %)	Error (%)
Cu	29	K-series	3666	66.79	70.98	42.69	2.8
C	6	K-series	918	15.05	15.99	50.87	4.3
Fe	26	K-series	1055	6.75	7.17	4.91	0.8
Os	76	L-series	127	5.24	5.57	1.12	2.8
Al	13	K-series	68	0.28	0.29	0.42	0.4
Total:				**94.10**	**100.00**	**100.00**	

Figure 6. Iron and aluminium compositional data from the second run (sample mounted on copper holder).

Data was also taken from a simple iron foil that was sputter deposited using the same methods as described above without co-deposition of aluminium. In this case it was observed that there was a similarly strong copper signal from the target holder compared to the iron signal. The relative copper and iron data was similar to that seen in figures 5 and 6.

Results

The results in figure 6 (neglecting possible effects caused by the relatively dominant copper signal) show a ratio of iron to aluminium to be 4.91% to 0.42%. The corrected percentage of 7.88% aluminium (relative to iron) was acceptable to the user group and was subsequently used in their experiments.

References

1. M. Edwards *et al.*, *Phys. Rev. Lett.* **97**, 035001 (2006). *'Opacity Measurements of a Hot Iron Plasma Using an X-Ray Laser.'*
2. K. Seshan (Ed), *'Handbook of Thin-Film Deposition Processes and Techniques'*, 2nd Edn, Ch 8.
3. A. Furuya and S. Hirono, *J. Appl. Phys.* **68**, (1) 304, *'Target Magnetic Field Effects on Deposition Rate in RF Magnetron Sputtering.'*