CASE STUDY

Powder Degradation in Serial Production

The analysis of powder chemistry after repeated Additive Manufacturing builds was experimentally carried out for two material used in Selective Laser Melting. Two studies were undertaken, the first using Aerospace material In718 and the second medical grade Ti-6Al-4V.

For the Aerospace study a Realizer SLM 250 was employed and was equipped with a 200W fiber laser and works in a protective argon atmosphere with an oxygen content limited to a 0.2%. In the second study the equipment used was a Renishaw AM250 which also has a 200W laser and similar processing atmosphere.

The powder used for the experiments was Gas Atomized In718 and Plasma Atomized Ti-6Al-4V, both having a particle size optimized for the respective machine tools but nominally less than 50 microns.

In718 Degradation on a Realizer SLM 250

To study the effect of powder reuse after processing, a specific methodology was developed. It consists of an iterative process with several steps (see Figure 1).

First, after all the powder is loaded into the SLM machine, test samples were produced. The second step requires removing all powder from the build chamber sieved to remove coarse particles generated during the melting process. Before and after sieving, a powder sample was taken. The powder is then subject to a drying process, where another powder sample is collected. Finally, once the powder has been sieved and dried, it is loaded into the SLM machine to be used in a further build. This process is repeated for 14 builds in total.

FIGURE 1

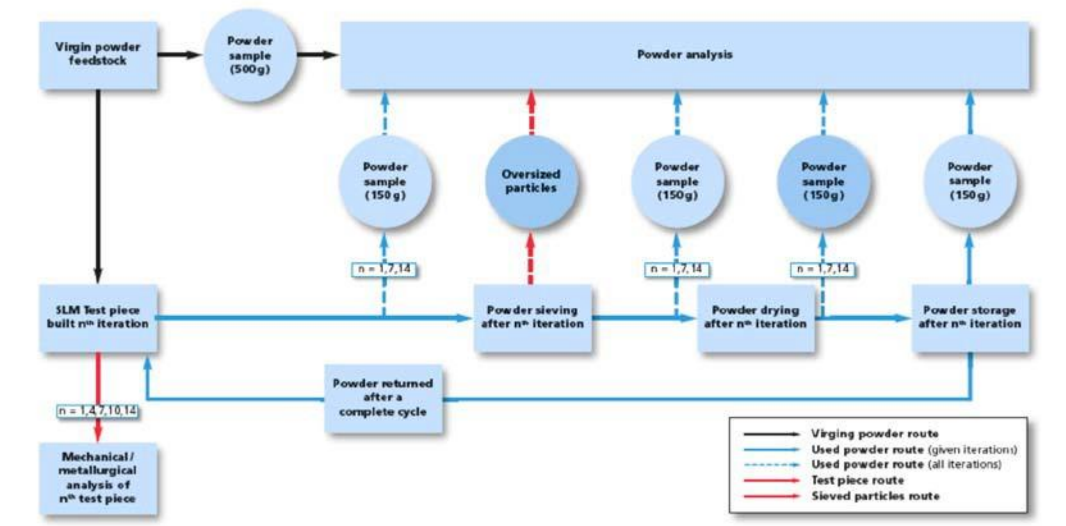

Figure 1. Workflow for experimental SLM builds employed to test the effect of powder reuse

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Chemical analysis was performed on the powder samples taken, Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) was used to test the bulk chemical composition, which revealed no significant change in chemistry.

The Oxygen content was analyzed using Inert Gas Fusion (IGF), samples taken after the first build, after sieving and after drying showed no changing in Oxygen content in the In718 powder, however powder tested after subsequent builds showed an increase in oxygen with each build.

This is illustrated in Figure 2 where an increase of 20 ppm over 14 builds is observed.

A retained sample of the initial powder was tested for Oxygen content after storage in a sealed container at ambient conditions and no significant change was observed.

Further builds were carried out and tensile specimens were produced. The test results can be seen in Figure 3, after 25 builds there is a significant increase in Ultimate Strength and reduction in ductility from 20% to approximately 13%.

FIGURE 2

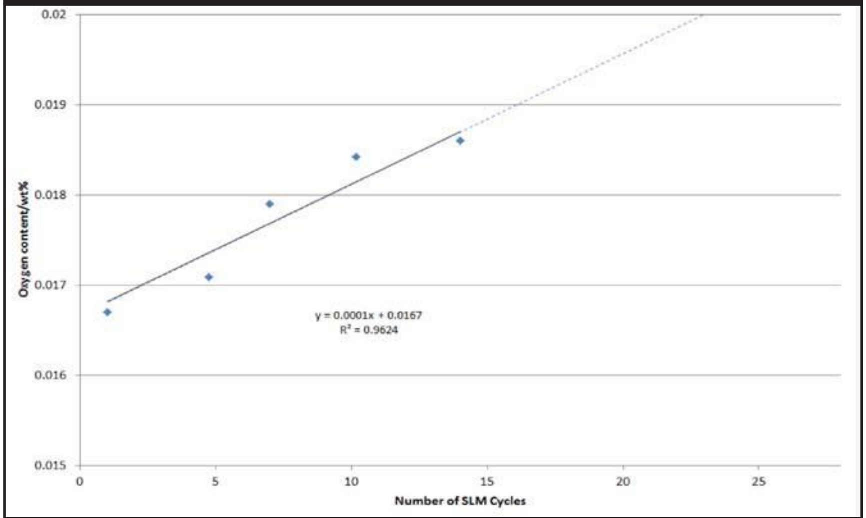

Figure 2. Oxygen content in In718 powder after repeated use in SLM builds

FIGURE 3

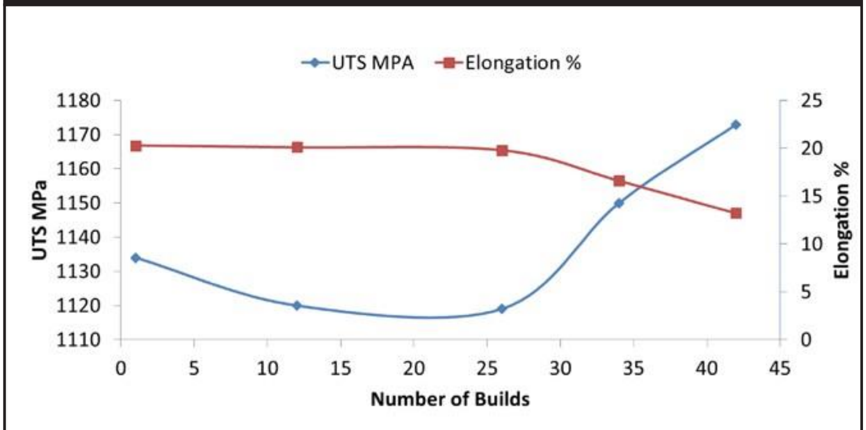

Figure 3. Effect of number of builds on mechanical properties of In718 tensile specimens produced by SLM

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Ti-6Al-4V

During the Titanium trial two powder lots were used. In the first, 10 builds were carried out following similar procedure to that described in Figure 1.

The effect of powder reuse is shown in Figure 4, over 10 builds the oxygen content increased by 200ppm, the initial powder chemistry met the requirements of ASTM B348 Grade 5 (0.2%max Oxygen) but not those of Grade 23 (0.13%max Oxygen).

Assuming the same gain would be repeated in subsequent builds the powder would not conform to ASTM B348 after approximately 20 builds.

Figure 5 shows SEM images of the powder after repeated use, re-melted particles and agglomerates are observed in the used powder.

A second study was conducted using Grade 23 powder over a much longer time frame. After a period of heavy use a blending procedure was undertaken to mix used powder with virgin powder to establish the effect on oxygen content when the powder lot is 'refreshed' by blending.

FIGURE 4

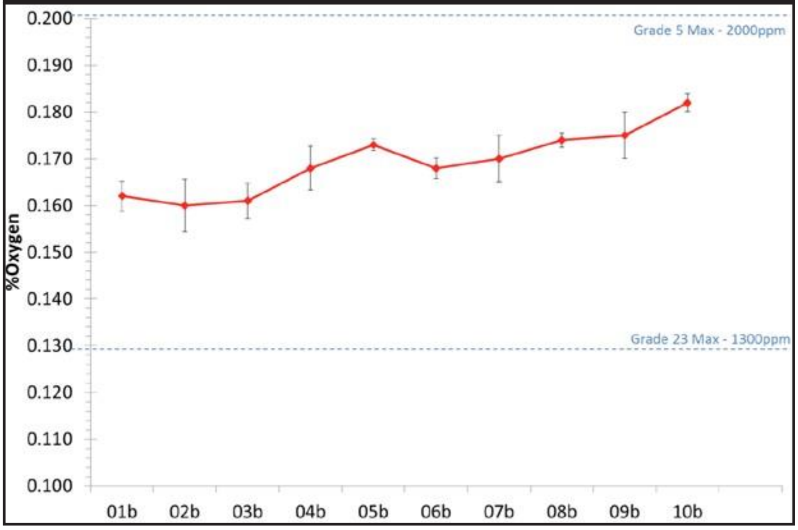

Figure 4. Effect of number of builds on oxygen content of Ti6Al4V powder used for SLM

FIGURE 5

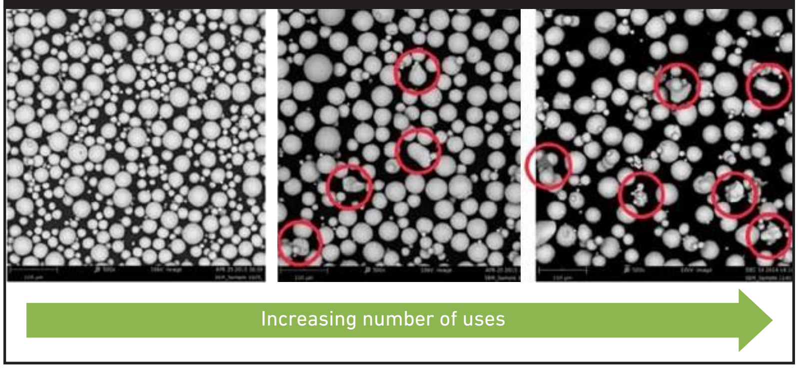

Figure 5. SEM images of virgin powder and used powder after several SLM builds

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Figure 6 shows the measured oxygen content over this time period which is split into several 'phases' (numbered 2-7 on the horizontal axis).

Key points of note in the different phases identified in Figure 6:

Phase 2 - heavy use of power (>20 builds) between two samples shows Oxygen pick up of approximately 200ppm. Further use into phase 4 shows powder exceeding the Grade 23 limit.

Phase 4 - reduction in Oxygen content due to blending of used powder with virgin.
Phase 5 and 6 - usage of powder employing a 'topping up' or 'refresh' strategy of powder during AM part production. Used powder was replaced with virgin powder after each build.

Phase 7 - previously blended powder was used without 'refreshing' procedure and Oxygen pick-up is again observed.

FIGURE 6

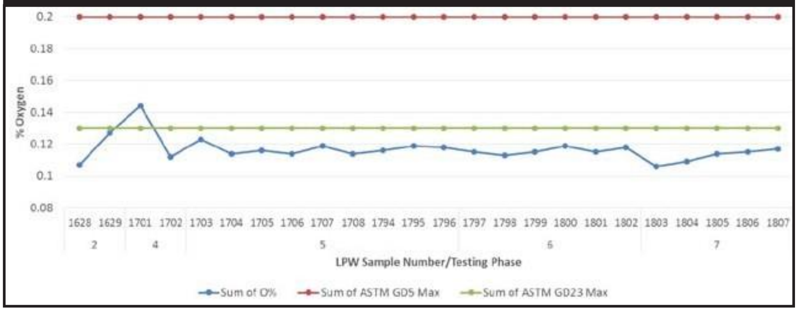

Figure 6. Oxygen content of Ti6AL4V powder used over a prolonged period and many SLM builds

Conclusions

In both In718 and Ti6Al4V there is an increase in Oxygen content with repeated use of the powder in SLM. In In718 it was shown that this results in significant reduction in ductility and increase in UTS.

It was also shown that blending used powder with virgin powder with lower Oxygen content will help to maintain the average value through extended manufacturing runs.