

# Vacuum vs non-vacuum melted gas atomised powders

# 09

### Gas atomisation

The melting of bulk material feedstock during gas atomisation (GA) typically takes place in a crucible. GA can be in open air, under an inert gas blanket or in vacuum conditions. For applications where the mechanical properties need to be tightly controlled, the level of gaseous impurities in the feedstock powder is key to the integrity of the final built part.

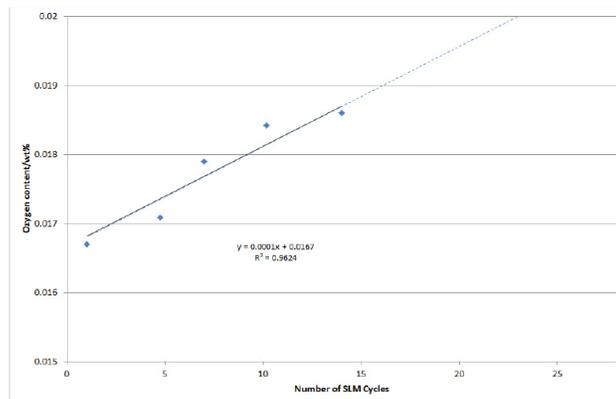
Studies of metal powders, using different techniques for melting during gas atomisation, have shown that open air techniques generate powders with the highest level of gaseous impurities. Atomisation techniques using an inert gas blanket during the melting stage show an improvement but still may not deliver the required low level of oxygen. The lowest levels of oxygen and nitrogen are achieved using vacuum atomised powder.

Superalloys, which can be used at high temperatures, benefit from several key characteristics including excellent mechanical strength, resistance to thermal creep deformation, resistance to corrosion or oxidation and good surface stability. Nickel-based superalloys are suitable for applications that require excellent corrosion resistance and advanced mechanical properties over a large range of temperatures.

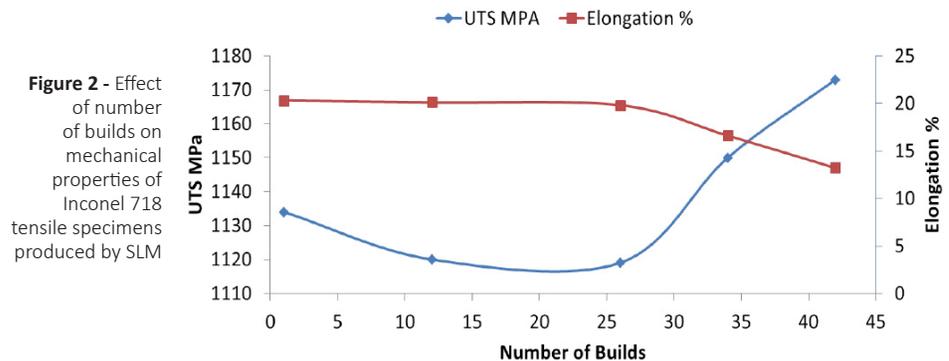
### The effect of oxygen and other impurities on the mechanical properties of nickel-based superalloys

During metal Additive Manufacturing the quality of the metal powder degrades after repeated use through multiple manufacturing cycles, potentially affecting the performance of the built component. This is demonstrated in Figure 1 for Ni-based alloy Inconel 718, by the increasing levels of oxygen in the powder measured after repeated build cycles.

Measuring the mechanical properties of the built parts shows that after 20 to 25 SLM build cycles, as the oxygen level is expected to exceed 200 ppm, the mechanical properties start to degrade. Similarly, Figure 2 shows the decrease of elongation and increase of UTS occurring after 20 to 25 cycles, whereas below that level, when the oxygen levels are lower, the mechanical properties remain reasonably stable.



**Figure 1 -** Oxygen content in Inconel 718 powder after repeated use in SLM builds



**Figure 2 -** Effect of number of builds on mechanical properties of Inconel 718 tensile specimens produced by SLM

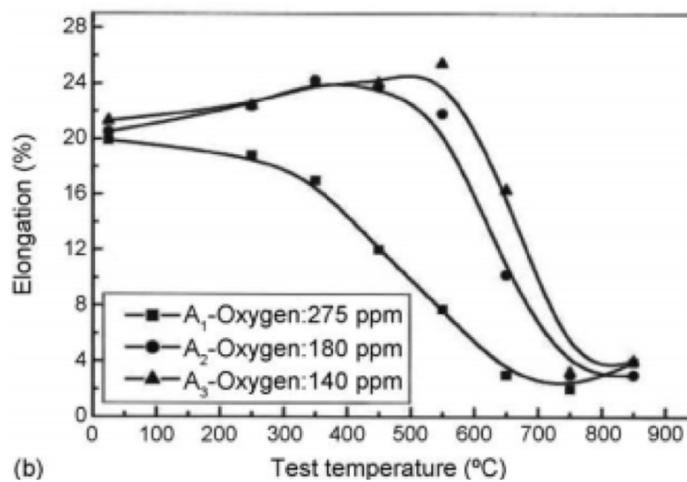
How oxygen and other gaseous contaminants affect the microstructures and mechanical properties of AM components is not well documented. Oxygen at low concentrations is not considered detrimental, but at a content above 200-300 ppm is shown to affect the mechanical properties such as elongation and rupture life. Nitrogen and hydrogen are considered harmful even in minute additions. Examples of detrimental and beneficial trace elements and impurities for Ni-based superalloys is shown in Table 1.

**Table 1** - Classification of impurities and trace elements in nickel-based superalloys<sup>1</sup>

Detrimental Elements	Examples
Residual gases	O, H, N, Ar, He
Non-metallic impurities	S, P
Metallic or metalloid impurities	Pb, Bi, Sb, As, Se, Ag, Cu, Ti, Te
Beneficial Elements	Examples
Refining aids	Ca, Mg, Ce, La
Minor and ppm alloying additions	B, Zr, Hf, Mg, C
Alloying addition up to 1.5%	Hf, Zr

Figure 3 shows the results of a study on the effect of oxygen levels on the mechanical properties of ‘as-HIPed’ 718 superalloy tensile samples. Elongation of the two samples manufactured from powder with an oxygen level below 200 ppm is quite similar, although the sample with the lower oxygen level does demonstrate a slightly superior performance for ductility/elongation. When the oxygen level of the feedstock powder is above 200 ppm, 275 ppm in this case, a significant deterioration in mechanical properties is observed.

**Figure 3** - Elongation of as-HIPed superalloy 718 with different oxygen levels as a function of temperature<sup>2</sup>



<sup>1</sup> More information and details about the effect of each of these elements can be found in ‘Impurities and trace elements in nickel-based superalloys’ by R.T Holt and W. Wallace (<http://www.tandfonline.com/doi/abs/10.1179/imtr.1976.21.1.1>).

<sup>2</sup> More details about this effect can be found in the ‘Effect of oxygen content of powder on microstructure and mechanical properties of hot isostatically pressed superalloy Inconel 718’ by Rao, G. Appa, M. Srinivas, and D. S. Sarma.



# LPW TECHNOLOGY

## CASE STUDY 09: VACUUM VS NON-VACUUM MELTED GAS ATOMISED POWDERS

Table 2 shows the effect of oxygen content on rupture life on HIPed and heat-treated samples which have been subjected to 690 MPa stress at 650°C. It can be concluded that the oxygen content has a significant effect on the mechanical properties of the built material. For HIP processing this is well documented and is attributed to the formation of prior-particle boundaries (PPBs) accompanied by stable oxides ( $Al_2O_3$  and  $TiO_2$ ) as well as brittle metal carbides (Nb, Ti)C. Although the mechanisms are different during the AM process, an increased oxygen level also appears to equate to inferior mechanical properties.

### Oxygen and other gaseous impurities levels from different manufacturing techniques

Table 3 shows some typical values for oxygen and nitrogen content in nickel-based superalloys from different production methodologies.

Open air techniques will generate powders with a relatively high level of gaseous impurities, producing components with inferior mechanical properties. Techniques using an inert gas blanket during melting are typically seen to generate powders with an oxygen content at the limit of 200 - 300ppm specifications.

**Table 2 -** Stress rupture properties of HIP and heat treated superalloy 718 as a function of oxygen content<sup>2</sup>

Alloy	Oxygen (ppm)	Test Conditions	Rupture Life (Hours)	%EL
(A <sub>1</sub> )	275	650°C/690 MPa	27	2.1
(A <sub>2</sub> )	180	650°C/690 MPa	84.5	4.5
(A <sub>3</sub> )	140	650°C/690 MPa	116.1	6.0
AMS 5662J specification for wrought alloy 718	-	650°C/690 MPa	23 (minimum)	4.0 (minimum)

**Table 3 -** Typical gaseous impurity levels for nickel-based superalloys with different production methodologies

	Open Air	Inert Gas Blanket	Vacuum
Nitrogen (wt%)	0.06-0.09	0.02 – 0.04	0.005 – 0.015
Oxygen (wt%)	0.06-0.09	0.03 – 0.04	0.01 – 0.025

### Conclusions

It is important to understand how the different melting techniques in gas atomisation powder production impact on the level of impurities, particularly oxygen and nitrogen, in the processed powders. This has been shown to have a detrimental effect on the microstructures and mechanical properties of the final products, including elongation, ultimate tensile strength (UTS) and rupture life. To meet the O<sub>2</sub> content specifications for

critical applications such as aerospace components, vacuum atomisation may be the preferred atomisation technique.

LPW works closely with customers to ensure that the powder specification is appropriate to the needs of the application.