APPLICATION NOTE

Analysis of titanium powder for additive manufacturing with ARL EQUINOX 100 XRD and ARL QUANT'X XRF Systems

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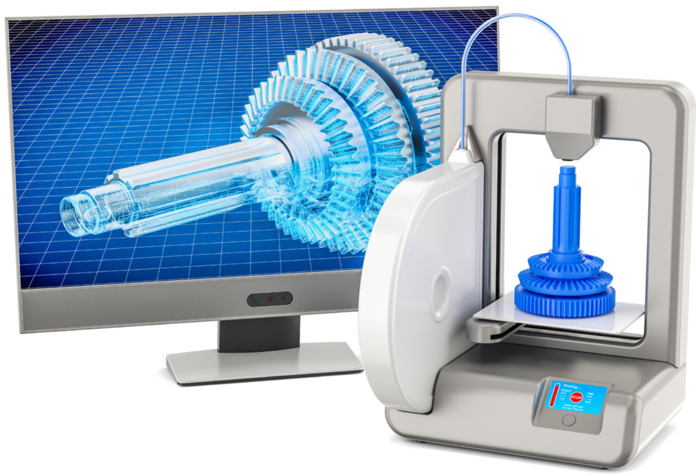

Introduction

Due to the fast-technological progression, the demand for complex metallic parts is growing rapidly. Using traditional processing methods for metals, there is a limitation in the complexity of parts. A newly evolving method to overcome this problem is additive manufacturing (also referred to as 3D printing), where parts are printed from metal powders as a "dye", layer by layer, using a laser or an electric arc to melt the powder. With this technique, the complexity of parts is unlimited, but the quality of the raw material must be controlled carefully to ensure the mechanical quality of the products. TC4 (Ti64, Ti-6Al-4V) is the most commonly used Ti alloy and is used, for example, in the aerospace industry or for implants and prostheses. Powders are produced by melt spraying of educts which can cause variations in composition or oxidation of the sprayed melt. This results in inhomogeneous elemental composition throughout certain batches of powders or contaminations with oxides and variations in crystallite size. This could have a negative impact on the mechanical quality of manufactured parts. The combined usage of X-ray diffraction (XRD) and energy-dispersive X-ray fluorescence spectroscopy (EDXRF) enables one to control oxidation, crystallite size (CS) and elemental composition in raw powders.

Instrument

The Thermo Scientific™ ARL™ EQUINOX 100 X-ray Diffractometer employs a custom-designed Cu (50 W) or Co (15 W) micro-focus tube with mirror optics. Such a low wattage system does not require external water chiller or other peripheral infrastructure, allowing the instrument to be easily transported from the laboratory to the field or between laboratories.

The ARL EQUINOX 100 Diffractometer (cf. Figure 1) provides very fast data collection times compared to other conventional diffractometers thanks to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time.

Figure 1: ARL EQUINOX 100 X-ray Diffractometer, fast, real-time and convenient.

The Thermo Scientific™ ARL™ QUANT'X Energy-Dispersive XRF Spectrometer uses a highly sensitive silicon drift detector (SDD) to discriminate between the energy of the incoming radiation and therefore can measure all elements between Na (Z=11) and Am (Z=95). It is equipped with a 50 W Rh or Ag tube which can be operated at voltages up to 50 kV. Conversion of spectra into elemental/oxide concentrations is achieved with the fundamental parameters (FP) based on the Thermo Scientific™ UniQuant Software standard-less package. The rugged and compact design as well as low demand on peripheral support make the ARL QUANT'X Spectrometer a perfect solution for industrial environments.

Experimental

For XRD measurements, samples of TC4 powder were measured in reflection geometry for 5 min under Cu-Kα radiation. Qualitative and quantitative analyses were carried out using MDI JADE 2010 with the ICDD PDF4+ database. EDXRF tests were carried out on powder samples – in a sample cup sealed with a 4-micron thick prolene film – under He atmosphere. The EDXRF semi-quantitative analysis results were obtained with the exclusive UniQuant Software.

Results

XRD analysis (c.f. Figure 3) of the TC4 sample clearly shows a Ti-Al alloy ($Ti_{0.85}Al_{0.15}$) and some $V_{11}O_{16}$ (c.f. Table 1). Usually Ti64 consist of α- (stabilized by Al) and β- (stabilized by V) Ti alloys. In the current sample, only the α-Ti phase is present, while β-Ti is completely missing as V is found as $V_{11}O_{16}$. The elemental composition taken from XRD yields 2.1 wt% V, which is already very close to the certified concentration of 3.5 - 4.5 wt% for TC4. Scherrer's equation yields a CS of 18.8 nm for the Ti-alloy and 31.4 for $V_{11}O_{16}$, which is very low compared to the specified particle size between 20 and 60 µm. As Scherrer's equation only yields the size of scattering domains, the particles might consist of several crystallographic independent crystallites, which stick together.

EDXRF analysis results obtained using UniQuant Software are shown in Table 2. As indicated by the XRD analysis, the main alloy elements are Ti, Al and V. In addition, Fe is detected at 2.5wt% as well as some Cr at 1.0wt%. Fe and Cr are normally not present in Ti64 at such concentration levels. Fe is typically present at less than 0.3wt% while Cr is at less than 0.1wt% if present at all. The presence of these elements indicates a cross contamination during the production of the powder. EDXRF offers a powerful solution as a QC/QA tool.

EDXRF also allows the detection of trace elements not readily accessible using XRD.

Figure 2: ARL QUANT'X EDXRF Spectrometer

Figure 3: Diffraction patterns of Ti64 measured with Cu-Kα radiation

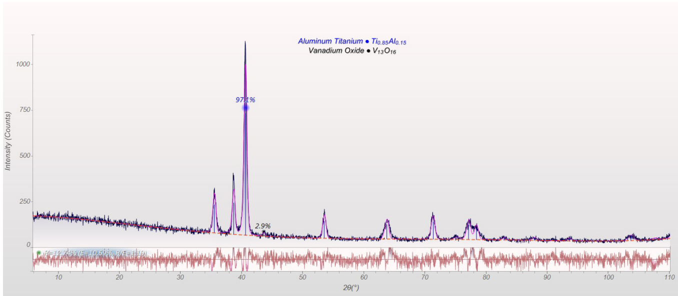

Table 1: Phase composition from Rietveld refinement.

Phase (in wt%)	Composition (in wt%)
$Ti_{0.85}Al_{0.15}$	97.1
$V_{13}O_{16}$	2.9

Table 2: EDXRF analysis results Ti64 sample.

Element	Conc. (wt%)	1-sigma (wt%)
Ti	83.63	0.19
Al	8.49	0.14
V	3.61	1.36
Fe	2.50	0.08
Cr	1.00	0.07
Mg	0.371	0.059
Ni	0.130	0.006
Mo	0.100	0.001
Mn	0.096	0.026
Co	0.035	0.015
Cu	0.018	0.002
Zr	0.016	0.001
Sn	0.006	0.001

Conclusion

The combination of XRD and EDXRF allows for the determination of elemental composition as well as speciation of TC4 powders. XRD reveals the crystallographic structure of TC4 and clearly indicates oxidation. MDI JADE 2010 combined with readily available databases make for a user-friendly tool to quantify the results. Though concentration estimates of major components are also possible with XRD, EDXRF offers a much more extensive and precise quantitative composition of the sample. The availability of standardless FP based semi-quantitative software such as the UniQuant Software avoids the need for suitable standards. Therefore, it is possible to detect trace elements, most likely due to cross contamination which might have negative influence on the material properties.

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