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Study of metal powders oxidation by means of Energy Dispersion Spectroscopy (EDS)

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Background incl. aims
Energy Dispersion Spectroscopy (EDS) is a very common technique for elemental composition characterization based on the capacity of specimens to produce characteristic X-rays when illuminated by an electron beam.
Different works have already described the possibility of using EDS for the determination of the thickness of metal and oxide thin film. Such approaches usually rely on the simulation of EDS spectra by means of Monte Carlo method-based algorithms.
The aim of this work is to apply EDS for the determination of the thickness of the oxide layer formed onto the surface of copper alloy powder during its storage in order to provide a tool to rapidly assess the "goodness" of the powder and its compatibility with the laser-based additive manufacturing processes.

Methods
Powder of CuAg 3.4 alloy was prepared via a gas atomization process (VIGA) and sieved to obtain a size distribution compatible with additive manufacturing processes by laser bed powder fusion (LBPF) technique.
The powder was then divided into two batches and stored for 1 month under different conditions. In particular, the first batch was stored under dry Argon whereas the second was stored at room conditions.

The chemical composition of the surface of a CuAg 3.4 alloy powder was investigated by means of Raman spectroscopy using a Renishaw InVia Raman confocal microscope using an excitation wavelength of 633 nm. The optical response of the powder was recorded in diffuse reflectance mode (incidence at 0°) using a Shimadzu UV-2600 UV-visible spectrophotometer equipped with an ISR2700 plus integration sphere.
EDS spectra were recorded using a Zeiss EVO 15 SEM equipped with a Peltier-cooled Oxford Ultim Max 40 EDS detector.

EDS spectra of CuAg 3.4 particles coated with different thicknesses of cupric oxide (CuO) layer were simulated using the WinMCXRay.

Results
From UV-Vis and Raman spectra, it was concluded that the inert sample developed a mixed Cu2O/CuO oxide layer of 5.4 nm thickness whereas the oxidized sample exhibited a thicker layer (15.3 nm) composed mainly of CuO.

Using the WinMCXRay tool, the EDS spectra of CuAg 3.4 particles coated with an oxide layer of different was simulated, and the γ was defined as the ratio between the x-ray count at 0.525 keV (Cu Kα) and 0.931 keV (Cu Lα).

EDX spectra of the two samples were collected under different beam acceleration tension conditions in the range from 3 kV to 12 kV, using a beam current of 800 pA.
After background removal, the value γ was calculated on the experimental spectra and the obtained value was compared with the simulated data to determine the oxide layer thickness t_CuO. For simplicity, we assumed that the oxide layer is composed of CuO only.

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We obtained a value of t_CuO=4.6 nm and t_CuO=16.1 nm for the inert and oxidized samples, respectively.

Conclusion

Energy dispersion spectroscopy was revealed to be a very useful and effective tool for assessing the extent of surface oxidation on metal powder. The oxide layer thickness evaluated by EDS are in good agreement with the estimation obtained from the UV-visible measurements and confirm the validity of the adopted procedure.
The possibility of measuring the oxide thickness without the need for more expensive and complicated instruments is of utmost importance in both academic and industrial contexts.

Keywords:

Metal powders, Energy Dispersion Spectroscopy

Reference:

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