SLS Symposium on

Microstructure investigation
Diffraction and scattering

Tuesday, June 12, 2018

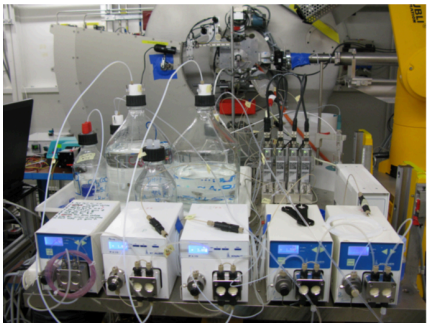
10:00 to 11:45, WBGB 019

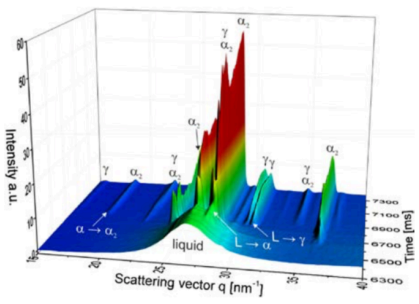
10:00 X-ray in-situ investigation of metals during Selective Laser Melting (SLM)
Samy Hocine, T. Maimaitiyili, D. Grolimund, S. Van Petegem, H. Van Swygenhoven

10:30 Chemical order 18 carat red gold alloys
Marina Garcia Gonzalez, N. Baluc, A. Díaz, T. Fischer, T. Connolley, S. Van Petegem, H. Van Swygenhoven

11:00 Coffee break

11:15 In situ synchrotron scattering studies on ACC colloidal suspension objects in solution
Ahmed S. A. Mohammed, A. Cervellino, A. Testino, A. Carino, M. R. Andalibi





X-ray in-situ investigation of metals during Selective Laser Melting (SLM)

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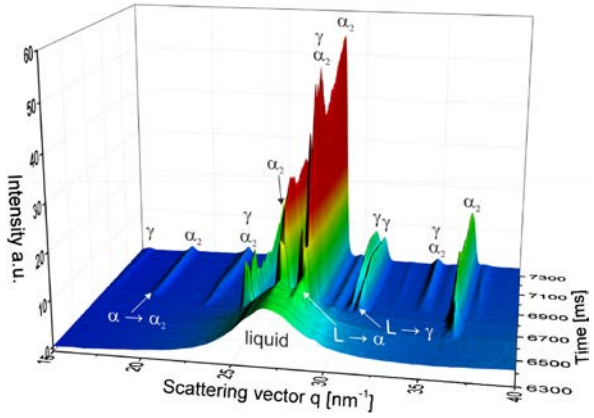

Figure 1: phase evolution in Ti-48Al during rapid solidification [1]

Selective Laser Melting (SLM) is a well-known process category in Additive Manufacturing (AM) in which thermal energy selectively fuses regions of a powder bed. Ex-situ characterization of SLM-made parts consists in exploring the microstructure for a certain range of parameter values (laser power, scanning speed, etc.). However, this is time consuming since a sample must be prepared for each set of parameters.

An attempt to track in-situ the rapid solidification of a Ti48Al sphere [1] has been made at the MicroXAS beamline of PSI. This experiment gives an overview of phase evolution depending on the cooling rate applied on the sample. However, the setup does not properly mimic the condition met during SLM since the sample boundary is a free surface.

To investigate process parameters for metallic materials in-situ with synchrotron X-rays, a miniaturized SLM device is under development at PSI. The design of the miniaturized SLM device is determined by the requirements for X-ray access and implementation at different beamlines of synchrotron light sources. In-situ X-ray diffraction/imaging during SLM will allow to obtain in depth understanding of the SLM process such as insight into the physical processes that occur during SLM, including the powder melting and solidification, phase formation in the melt-pool, the formation of pores and residual stresses.

[1] C. Kenel, D. Grolimund, J.L. Fife, V.A. Samson, S. Van Petegem, H. Van Swygenhoven, C. Leinenbach, Combined in situ synchrotron micro X-ray diffraction and high-speed imaging on rapidly heated and solidified Ti–48Al under additive manufacturing conditions, Scr. Mater. 114 (2016) 117–120. doi:10.1016/j.scriptamat.2015.12.009.

Chemical order 18 carat red gold alloys

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Introduction

Residual stress management is a major concern during the processing of 18 carat Au-Cu-Ag alloys. Depending on thermo-mechanical history and chemical composition residual stress causes reduced workability, shape distortions and eventually fracture. It is anticipated that a solid state order/disorder phase transformation forming $Au_{50}Cu_{50}$ precipitates lies at the origin the residual stress.

18 carat red gold is an age-hardenable alloy which hardens via chemical ordering. Above the critical temperature ($T_c \approx 350ºC$) the alloy has a chemically disordered FCC structure. However, below this temperature, chemical ordering takes place and a dispersion of harder nano-precipitates of $Au_{50}Cu_{50}$ phase starts to form. This causes a tetragonal distortion of the initial cubic lattice and induces strong misfit strains. The kinetics of the ordering process strongly depends on the initial state, e.g. the temperature at which previous disordering was done, cooling rate and stress state. This implies that the final microstructure in terms of grain size and ordered volume fraction will depend on the history of thermo-mechanical processing. Our study investigates how thermal parameters and plastic deformation affect the ordering kinetics and the morphology of precipitates, with the aim of understanding the link between precipitation and macroscopic properties.

Here, we present a combination of findings from 1) X-ray diffraction data collected at the I12 beam line in Diamond and P07 beam line in PETRA, 2) X-ray scattering data collected at the cSAXS beam line in SLS and 3) TEM observations at CIME in EPFL. Overall, we investigate the effect of plastic deformation and increasing vacancy densities on the ordering kinetics at RT, and the resulting microstructure. At Diamond we preformed scans across a deformed wire to look at the role of plastic deformation on the precipitation distribution, at cSAXS and PETRA we investigated the role of the cooling rate on the precipitation kinetics and with TEM we confirm the precipitation distribution post-mortem.

In situ synchrotron scattering studies on ACC colloidal suspension objects in solution

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The nature of formed entities at the early stage of the precipitation pathway of calcium carbonate in supersaturated solutions is a matter of debate in literatures. Classical scattering methods such as Wide-Angle X-ray Scattering (WAXS) and Small-Angle X-ray Scattering (SAXS) techniques were employed for the investigations of amorphous calcium carbonate (ACC) entities formed from supersaturated solutions. SAXS experiments were conducted at the material science beamline (MS – X04SA) of SLS at PSI. We specially designed contactless devices for the measurements, generating stable liquid jets from supersaturated solution with controlled *pH* and saturation, using four HPLC pumps and flowmeters for mixing reacting solutions in the jet (NaOH, $CaCl_2$, $NaHCO_3$, H_2O) as shown in figure 1(left). Thermal baths helped control *T*. Scattering data were collected using a Mythen II detector. The data were modeled using parametric statistical models providing insight about the size and shape distribution of denser matter in the liquid jet. A representative example for the mass distribution of the investigated entities is shown in figure 1 (right).

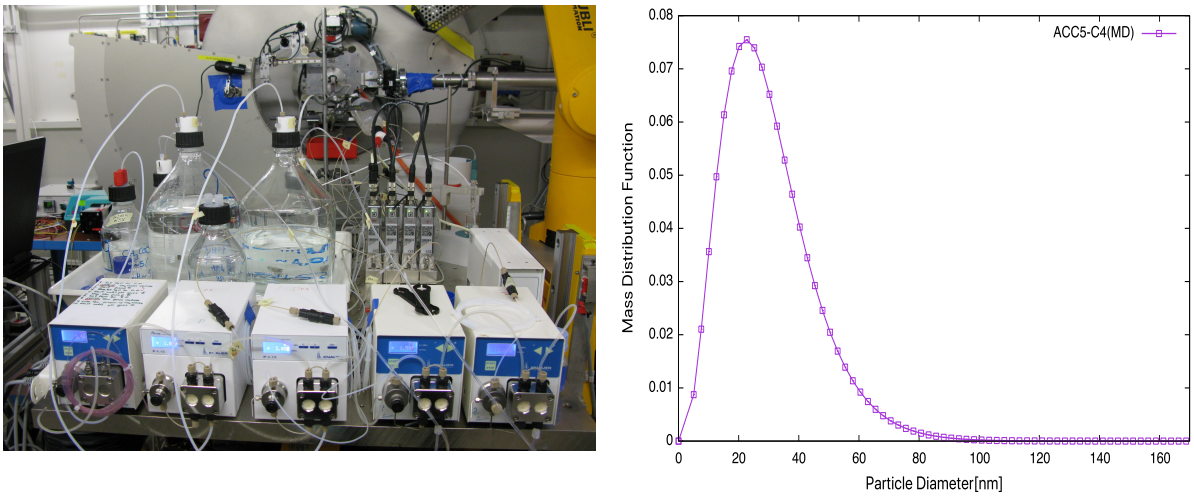

Figure 1. (Left) HPLC pumps delivering precursor solutions to a mixer and a liquid jet is generated. (Right) The mass distribution obtained for one of the supersaturation levels (S =1.46).