

Solidification Kinetics of an Oxide Weld Slag Utilizing SEM and LSCM Imaging

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Slag formation on solidifying weld pools strongly influences both the physical properties and chemical composition of the weld metal. This work focuses on characterization of a multi-component oxide (TiO₂-CaO-SiO₂-MgO) slag commonly used in different flux-based arc welding processes. Analysis was done using a Scanning Electron Microscope (SEM) in conjunction with Energy Dispersive Spectroscopy (EDS) to analyze the resulting slag microstructure and composition. A Laser Scanning Confocal Microscope (LSCM) was also employed in order to observe the slag solidification kinetics and the melting temperatures of observed phases.

Welds were made with electrodes of two different compositions, C1 and C2, which have intentional additions of fluorspar (CaF₂) and alumina, respectively. The slag was subsequently prepared for microscopy [1]. The SEM work was done with a FEI Helios Nanolab™ 650 operating at 20kV. Low magnification BSE images were taken in order to characterize the volume fraction of each phase in the microstructure, while higher magnification images allowed for EDS of individual phases as shown in Figure 1. By combining each phase's volume fraction with its composition, it was possible to find the liquid-slag composition before each phase of solidification (Figure 2).

The LSCM analysis was conducted on an Olympus FV1000 filter-based confocal scan head coupled to an Olympus BX62 upright motorized microscope. Slag samples were crushed and placed in a platinum crucible inside a Linkam TS1500 hot stage, heated to 1500°C in laboratory air, held for one minute, and cooled at 100°C min⁻¹. A video recorded the heating and cooling, which showed the melting of the slag, wetting behavior, and solidification temperatures of individual phases (select images shown in Figure 3). Using the known frame rate and a controlled heating rate, temperature was superimposed on the images using ImageJ software. The tests were operated with the shortest wavelength laser available (405 nm, violet) as optical resolution is directly proportional to wavelength and wavelength filters were used to eliminate the interference from incandescence, which severely impedes conventional optical imaging at similar temperature ranges [2].

Analysis of the data showed that in this slag system, the solidification kinetics are primarily driven by titanium composition, with titanium-rich (50%) primary dendrites (D₁) forming first, followed by 30% Ti secondary dendrites (D₂), and a Ti-deficient (<8%) glassy (G) phase. From the EDS results, it was found that both additions preferentially segregate to the glassy phase. However, the data suggests that the addition of fluorspar in C1 allows for greater formation of crystallinity than the C2 composition, at the expense of the glassy phase. As the glassy phase is the last to solidify, and the liquid-phase controls the wetting action on the steel, this data suggests that additions of alumina may improve wetting characteristics of slag, and consequently provide better atmospheric protection, resulting in improved weld properties.

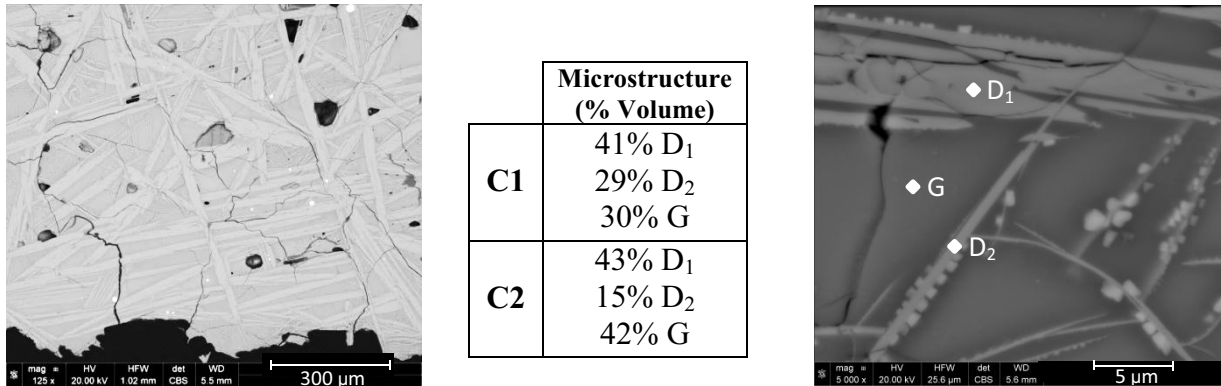


Figure 1. Typical SEM images and phase composition by volume of microstructure of C1 and C2.

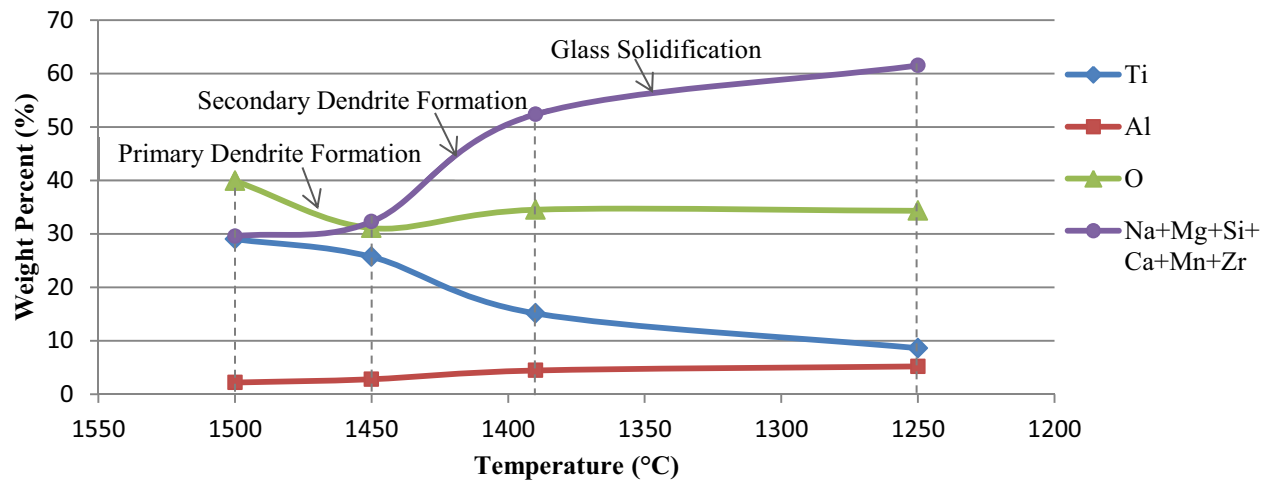


Figure 2. Solidification curve of C2 liquid composition, with phase formations marked.

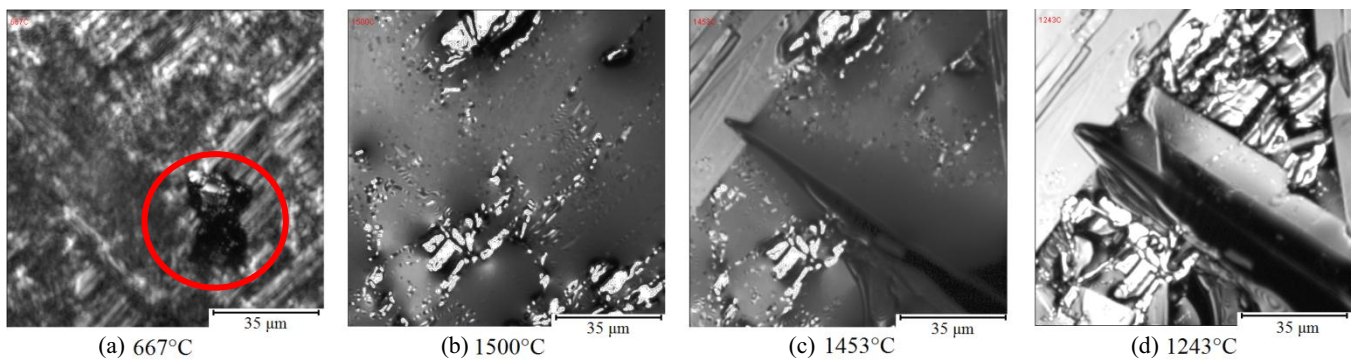


Figure 3. LSCM images of: (a) slag particle, (b) max wetting, (c) partial and (d) full solidification.

References:

[1] BK Narayanan, A Avishai, “Characterization of a Multi-Component Oxide Slag Formed Over a Ferritic Weld Metal using HAADF/STEM Imaging” *Microscopy and Microanalysis* (2012) pp 1668-1669.

[2] D Hovis, A Heuer, “The use of laser scanning confocal microscopy (LSCM) in materials science.” *Journal of Microscopy*, **240** (2010), pp 173-180.