

Title: "Characterization and modification of powders used to make aluminium-based metal foams"

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Abstract

Aluminium-based metal foams made by the powder metallurgical route provide scope and two objectives for this thesis. The manufacturing route consists of admixing to an aluminium powder alloy a blowing agent, commonly 0.5-1.0 wt.% of pre-oxidized TiH₂. The powder mixture is hot-compacted to produce foamable precursors. Later the foaming process is initiated by heating the precursor material.

The first objective of the thesis is to evaluate whether hot-compaction of powders under vacuum is a good strategy to produce foams more regularly distributed in pore sizes than traditional hot-compaction in air. For this, AlSi11 foams containing TiH₂ and free of blowing agent were produced both under vacuum and in air and compared. The foaming behaviour was studied in-situ by X-ray radioscopy, the hydrogen evolution was followed by mass-spectrometry, and the resulting porous structure was characterized by X-ray tomography. It was found that hot-compaction under vacuum led to better consolidation of foamable precursors by improving the degree of metallic bonding between aluminium particles which retarded hydrogen losses before melting and reduced the growth of cracks before melting. The result was a larger and more homogeneous expansion and a more regular pore size. The foam made from vacuum-pressed powders had 37% larger volume than the one made from air-pressed powders and twice narrower pore size distribution. Both are measures of the improvement introduced by hot-compaction under vacuum.

As TiH₂ is the most commonly used blowing agent to produce aluminium-based metal foams, the second objective is devoted to elucidate whether there is a relationship between the phase transformation sequence and the regimes of hydrogen release during decomposition. The phase transformation sequence of untreated and pre-oxidized TiH₂ was studied in-situ by energy dispersive X-ray diffraction using synchrotron radiation and correlated with the release of H₂ gas measured by mass spectrometry. Partially decomposed untreated powders were additionally investigated by electron microscopy to determine the spatial distribution of phases and verify a core-shell model. In untreated powders the starting phase is δ -TiH₂, in which the Ti atoms form a fcc sub-lattice. As temperature increases two other structures which dissolve H in interstitial solid solution appear: hcp α -Ti and bcc β -Ti. Untreated TiH₂ transforms according to: $\delta \xrightarrow{432^\circ\text{C}} \delta + \alpha \xrightarrow{514^\circ\text{C}} \delta + \alpha + \beta \xrightarrow{536^\circ\text{C}} \alpha + \beta \xrightarrow{702^\circ\text{C}} \alpha$, heating at 10 K.min⁻¹ under Ar flow. Volume fractions of phases estimated from integrated X-ray diffracted intensities were used to construct radial compositional diagrams vs. temperature assuming spherical particles. The α phase appearing forms an outer shell around a δ -core due to the Ar flow which favours the nucleation of α at the particles surface. The H₂ release from the untreated TiH₂ decreases when the thickness of the α shell increases. After oxidation pre-treatment at 480 °C for 3hs in air, a core-shell structure forms consisting of a δ -core an intermediate layer of Ti₃O and an outer-shell of TiO₂. The pre-treatment modifies the phase transformation sequence. The release of H₂ from pre-treated powders is controlled by the thickness of the outer TiO₂ layer. Above 535 °C, Ti₃O transform into hcp α -Ti which reduces TiO₂. The phase transformation temperatures are retarded by increasing the heating rate and anticipated if powders are compacted.