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Combustion foaming of Al-Ti intermetallics studied *in situ* by fast X-ray radiography

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Abstract

Combustion foaming of Al-Ti intermetallics was observed *in-situ* by X-ray radiography at the Technical University Berlin, Germany, and at the experimental station ID-19 of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The acquisition rates were 6.7 fps and up to 500 fps, respectively. This allowed for observing in detail the internal structure development during combustion foaming of extruded Al-Ti powder compacts made with Al/Ti molar ratios of 3, 4 and 7 and containing 10 and 20 vol.% exothermic agent (Ti + B₄C). At the beginning of the foaming process, μm-sized pores were observed. Foam shrinkage and pore coarsening started immediately after the combustion process. Specimens made with an Al/Ti ratio = 7 showed pore coarsening and a longer solidification time. Specimens with 10 vol.% exothermic agent experienced less volume expansion and pore coarsening than specimens with 20 vol.% exothermic agent. The specimens with an Al/Ti ratio = 4 contained fewer micropores than the one with an Al/Ti ratio = 3.

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1. Introduction

Combustion synthesis foaming is a promising process for making high-melting point metal foam in a short time by inducing a combustion reaction in compacted elemental powders (Inoguchi et al. (2009), Kobashi et al. (2010)). The kinetics of the process that comprises the ignition stage, pore growth, pore coalescence and solidification could not be explored in sufficient detail up now due to the lack of methods to observe *in-situ* the highly exothermic and extremely rapid reaction.

This study reports on details of combustion foaming of Al-Ti intermetallic compounds obtained by *in-situ* X-ray radioscopy (García Moreno et al. (2004)) and comprising a real-time observation of expanding foams with both high spatial and time resolutions (García Moreno et al. (2008)).

2. Experimental procedure

2.1. Preparation of precursor

Aluminum (<45 μm , 99.99 %), titanium (<45 μm , 99.9 %) and B_4C (<0.5 μm , 99 %) powders were used in this study. The molar blending ratios between Al and Ti were fixed to 3, 4 and 7 (Kobashi et al. (2010)). The amount of exothermic agent (B_4C) was adjusted such that 10 or 20 vol.% ceramic particles (TiC , TiB_2) were formed in the Al-Ti foam specimen. The blended powder was then hot-extruded at 673 K to rod-shaped precursors of 10 mm diameter. Cylindrical sections of 10 mm length were cut from the rods and used for X-ray radioscopy. Additionally, samples of $5 \times 5 \times 2 \text{ mm}^3$ size were prepared for synchrotron radioscopy experiments.

2.2. X-ray radioscopy scanner

The samples were analyzed in an X-ray scanner built for this purpose (García Moreno et al. (2008)). The cylindrical specimens were placed into a disposable, square steel section closed with an Al-foil in the X-ray direction. They were heated by a resistive carbon heating plate from below, protected by a Si_3N_4 coating. The entire setup was placed inside a X-ray transparent insulating box. The section protects the heating plate from the combustion reaction and helps reducing temperature losses close to the sample, so that the system can reach the ignition temperature in the desired temperature window. The samples were heated to 673 K in 90 s in the first step and held in this condition for 120 s to homogenize the furnace temperature. In the second step, the temperature was further increased to 1173 K to trigger combustion foaming. Radioscopic images of the process were recorded at a rate of 6.7 fps.

2.3. X-ray synchrotron radioscopy

The experimental station ID-19 of the ESRF in Grenoble (France) was used for fast white beam synchrotron radioscopy. The specimens were placed on the heating plate directly which works well for most of the samples. The temperature was first held at 623 K for 90 s and then increased to 1173 K to induce foaming. A LuAG:Ce scintillator in combination with a Dimax camera was used for detection. An optical magnification of 4 gave rise to an effective pixel size of 2.7 μm and a field of view of $5.4 \times 5.4 \text{ mm}^2$. The exposure time was 2 ms and the corresponding acquisition rate 500 fps. Phase contrast significantly contributed to the image quality.

3. Results and discussion

The combustion reaction always begins at the bottom of the specimen as this part is closer to the heating plate and then propagates rapidly through the rest of the sample. In the specimens containing 10 vol.% of the exothermic agent, less expansion and pore coarsening than in the specimen with 20 vol.% was found.

Fig. 1 shows the results of the X-ray observation on the specimen made with an Al/Ti ratio = 4 and 20 vol.% exothermic agent. The specimen expanded rapidly as the combustion reaction proceeded and immediately shrunk

after maximum expansion had been reached. However, the pores in the specimen continued to coarsen until total solidification of the specimen occurred more than 3 s later.

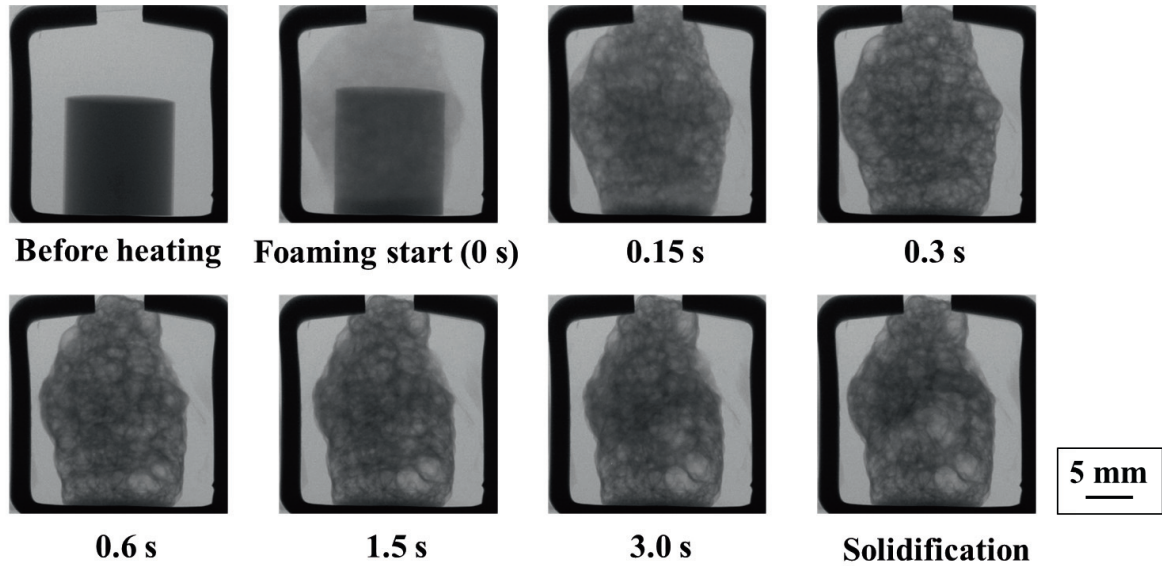


Fig. 1. X-ray image sequence of the specimen made with a Al/Ti ratio = 4 and containing 20 vol.% B₄C.

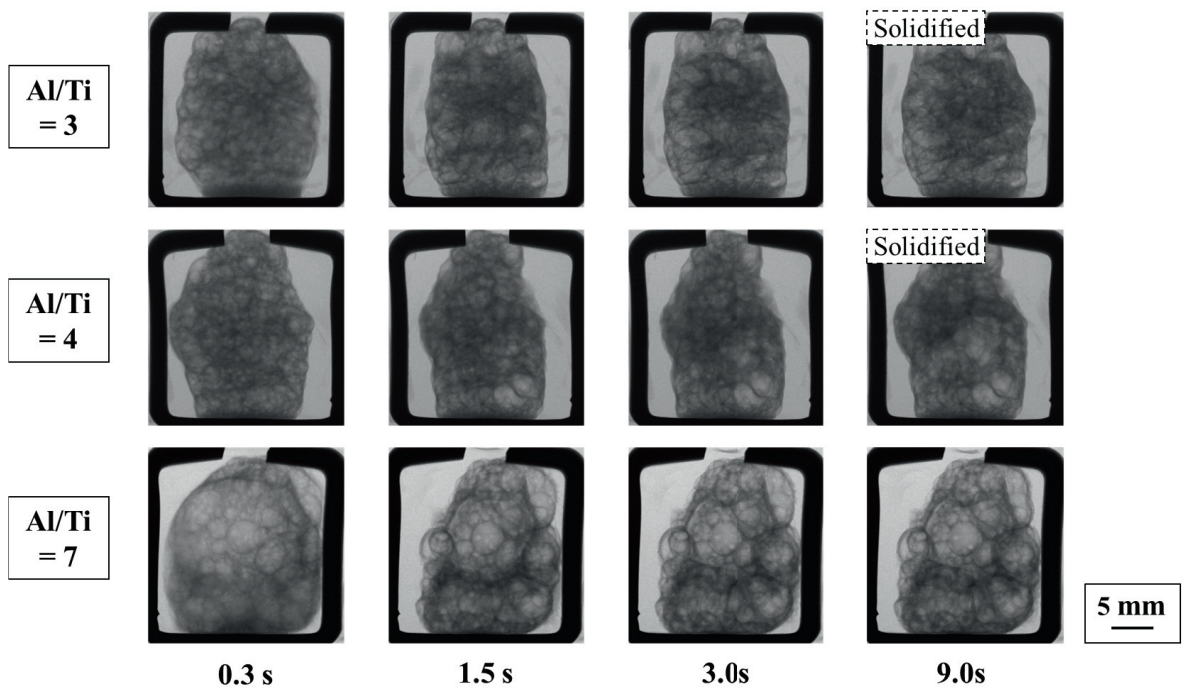


Fig. 2. X-ray image sequences of the specimen made with Al/Ti = 3, 4 and 7 and 20 vol.% exothermic agent.

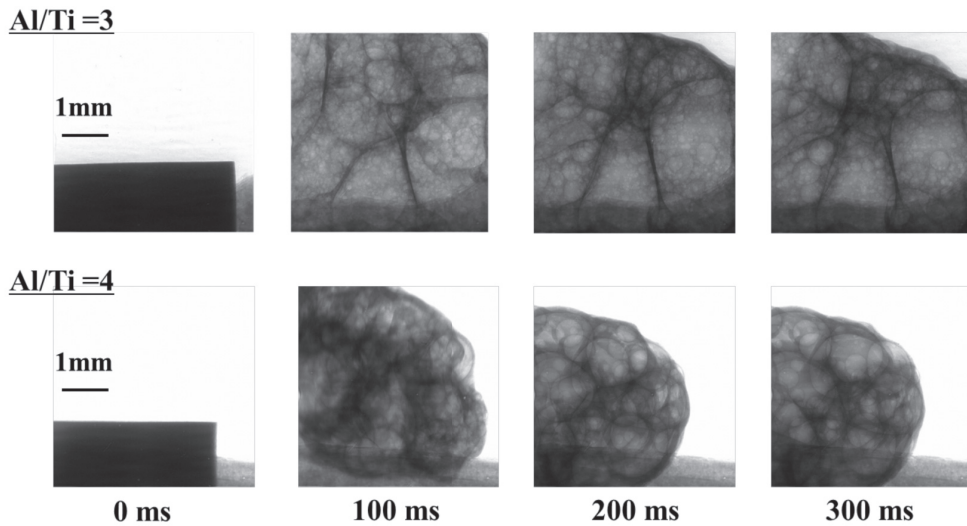


Fig. 3. Synchrotron X-ray image sequence of specimens made with Al/Ti ratios = 3 and 4 and 20 vol.% exothermic agent. Images show $t = 0$ s, 100 ms, 200 ms and 300 ms after the initiation of foaming.

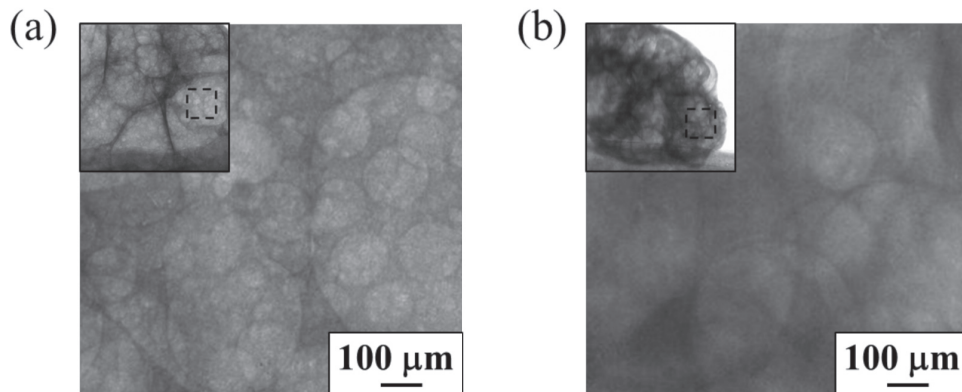


Fig. 4. High temporal and spatial resolution synchrotron X-ray images of specimens made with 20 vol.% exothermic agent 100 ms after initiation of foaming. (a) Al/Ti ratio = 3, (b) Al/Ti ratio = 4.

Fig. 2 shows images of an expanding specimen made with 20 vol.% exothermic agent and different Al/Ti ratios. For an Al/Ti ratio = 7, we observe the largest amount of pore coarsening and the longest solidification period of < 9 s. This is caused by a higher viscosity and lower solidification temperature of the liquid phase due to the greater amount of available Al. In this case, solidification finishes at the melting point of Al (933 K), much lower than that of Al-Ti intermetallics (Al_3Ti , 1613 K), and the viscosity of melt is increased by the presence of solidified intermetallics.

X-ray radioscopy with laboratory equipment can reveal the details of combustion foaming on a macroscale, but cannot make visible the early expansion phase and features on a microscale due to the lack of spatial and temporal resolution. Therefore, synchrotron radioscopy was applied. Specimens made with Al/Ti ratios = 3 and 4 and containing 20 vol.% exothermic agent were studied. Figs. 3 and 4 show some images extracted from synchrotron radioscopy sequences. In the specimen made with an Al/Ti ratio = 3, μm -sized pores in the foam structure exist as can be observed 100 ms after the initiation of foaming (Fig. 4). Such small pores are generated by the thin surface

hydroxides on Al powder particles, which play the role of a foaming agent (Kobashi et al. (2008)). The micropores remain stable for some time as can be observed in the images captured 300 ms after the initiation of foaming.

However, they begin to disappear as the larger pores begin to coalesce. In samples with an Al/Ti ratio = 4 such small pores could not be observed at all. One could either suspect that the micropores are absorbed by the larger pores during the first 100 ms, which would indicate that a higher Al/Ti ratio encourages immediate pore coarsening by combustion foaming, or that no such pores are created for high Al/Ti ratios.

4. Conclusions

Foaming of Al-Ti intermetallic compounds during the combustion reaction was observed by laboratory X-ray and synchrotron X-ray radioscopy. The effects of the Al/Ti ratio and the content of the exothermic agent on the foaming behavior were investigated:

1. Volume expansion and pore coarsening of specimens containing 10 vol.% exothermic agent was less than with 20 vol.% exothermic agent.
2. Higher pore coarsening and volume shrinkage were observed with increasing Al/Ti ratios.
3. Micrometer-sized pores were found immediately after the initiation of foaming in the foam derived from specimens made with an Al/Ti ratio = 3.
4. Micropores began to disappear as the larger pores began to coalesce.

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