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Effect of the interface bonding on the mechanical response of aluminium foam reinforced steel tubes

Lucio Bonaccorsi · Edoardo Proverbio · Nunzio Raffaele

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Abstract Aluminium foams have been recently proposed as filling reinforcements to improve impact behavior of hollow components used as protection systems in vehicles. In this study, aluminium foam filled stainless steel tubes have been prepared by directly foaming metal powder compacts inside the tubes. Attention was concentrated on the interface phenomena that characterize the core–shell interaction and the process parameters determining the metallurgical reactions between the two alloys. The formation of binary and ternary intermetallic compounds was observed at the aluminium/steel interface whenever the growth of the oxide layer on the foam surface in foaming was constrained. Compression tests of the reinforced tubes confirmed a maximized energy absorption in coincidence with the formation of the interface bonding. In those cases, extended foam intrusions into compressed tube folds were observed. The microstructural investigation revealed that in the transition zone the intermetallic layer strength was comparable to that of the foamed matrix.

Introduction

Aluminium and steel hollow profiles, such as tubes and columns, are usually added to the vehicle frame in order to reduce injuries to passengers during crashes by absorbing impact energy in deforming. Light metal foams have been recently proposed as filling reinforcements to improve the impact behavior of such protection systems. According to Ashby et al. [1], closed cell aluminium foams should excel as energy-absorbing materials and as stiffener for structural elements for their mechanical behavior in static and dynamic compression stresses. Moreover, the intrinsic low density makes aluminium foams particularly appropriate for transport industry applications and whenever weight saving becomes important. Several tests on bumpers, crash boxes and pillars usually made of hollow steel or extruded aluminium have shown positive results when aluminium foam reinforced [2]. The mechanical characterizations of foam-filled thin-walled columns, circular or not, have demonstrated that the specific (per weight) energy-absorbing potential improves in all crushing conditions, longitudinal and transverse, more than the mere addition of the energy contribution of each component [3–15].

Metal foam reinforced members could be obtained by three different methodologies: by direct foaming inside a hollow profile, by encasing the foam by casting (different casting methods are possible [10]) or by inserting in the hollow element a pre-foamed core [3]. In direct foaming, the metal is foamed inside a pre-existing shell, while by encasing the shell is cast around the foamed core. The production method has a great influence on the interface phenomena between the foamed core and the solid shell. The occurrence of an interface bonding increases the total energy absorbed by the reinforced element in compressive loads because of an "interaction effect" between the inner reinforcement and the containing component [5, 11]. Casting methods, in general, are not able to create an interface bond because of the continuous aluminium oxide skin covering the foam that prevents any surface reaction with the molten metal [10, 16]. In the cold insertion, the only possible join is obtained by friction or, alternatively, by gluing the foam to the shell [3, 6]. On the contrary, the in situ foaming, as in the direct filling process, should

L. Bonaccorsi (✉) · E. Proverbio · N. Raffaele
Department of Industrial Chemistry and Materials Engineering, Faculty of Engineering, University of Messina, Contrada di Dio, Messina 98166, Italy
e-mail: lbonaccorsi@unime.it

facilitate the interface reaction with the formation of a real metallurgical bonding, but the short time of the foaming process and the oxidization of the foam surface during the expansion could reduce significantly such a possibility.

The aim of the present work was to investigate the foam–shell interface formation and its influence on the mechanical behavior of reinforced stainless steel tubes prepared by direct foaming. Circular steel tubes have been filled with pieces of precursors obtained by powder compaction and directly foamed in a horizontal furnace where the foam evolution inside the tubes was evaluated by a self-made dilatometer. Interface reactions were preventively investigated by aluminium wetting tests in controlled atmosphere and the results compared with tube-filled interfaces. It was observed that direct foaming could produce an extensive bonding between the aluminium foam and the steel tube walls if the filling process is opportunely carried out to hinder the oxide layer formation. Finally, the reinforced tubes were mechanically tested by uniaxial compression and the effective contribution of the interface bonding in the mechanical response of the reinforced structure was clearly highlighted.

Experimental setup

Precursors preparation

Foaming precursors have been prepared by adding in a tumbling mixer aluminium (Ecka, AS 081 99.5% Al–45 μm) and silicon (GoodFellow, 97.5% Si–150 μm) powders together with 0.4 wt% titanium hydride (GoodFellow, 99.0% TiH_2–150 μm) used as blowing agent. Three different compositions were tested: (a) 99.6 wt% Al; (b) 92.6 wt% Al–7 wt% Si; (c) 96.6 wt% Al–3 wt% Si.

The powder mixtures were poured in a cylindrical mould (ID = 25 mm) and pressed by a cold uniaxial double pressure: after an initial compaction up to 160 MPa, the cylindrical die was reversed and the pressure was exerted on the reverse surface up to the final value of 279 MPa. All the prepared precursors got a green (relative) density over 98 wt%.

Foaming and tubes filling

Segments of 60 mm length (ID = 25 mm, th = 2 mm) of a stainless steel pipe (AISI 304) were cleaned at room temperature with trichloroethylene in an ultrasonic bath for 15 min, rinsed with distilled water and then pickled by HCl solution (1 M) for 15 min at room temperature with stirring. Following, the tubes were washed with 1 M NaOH solution, rinsed several times with distilled water and left drying at room temperature.

In the standard procedure, a cleaned tube filled with two precursor billets was set on a horizontally support and closed on the two sides by fixed lids. The system was placed in a horizontal furnace pre-heated at $T = 830$ °C and kept heating for 480 s. The sample was then quickly extracted from the furnace and cooled down by water spraying. In "controlled atmosphere" experiments, the furnace was equipped with a quartz tube containing the sample support, which allowed flowing nitrogen during the foaming process.

To monitor the filling process, the sample holder was modified and one tube side was closed by a piston put directly in contact with the billets (Fig. 1). The piston rod

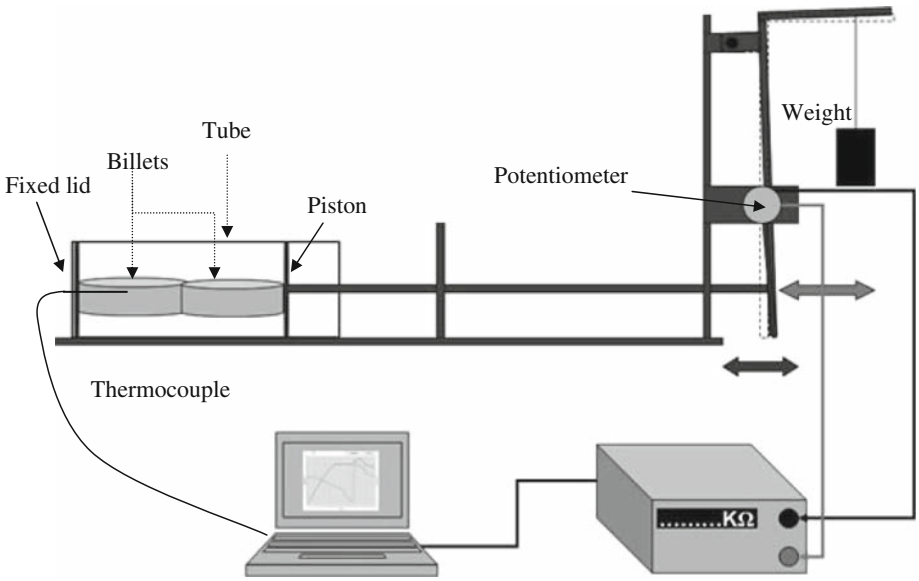


Fig. 1 Diagram of the system used to fill the steel tubes

was connected to a potentiometer to measure its displacement during heating. The system was placed in the horizontal furnace and heated until the expansion reached a reference value measured by the piston shift. The filling process was carried out in air. In a set of experiments, the piston rod was loaded by a weight (Fig. 1) so that the piston exerted a force $F = 2$ N to constrain the foam expansion inside the tube.

Wetting tests

A preliminary study of phenomena occurring at the interface between the aluminium foam and the metal case was conducted by foaming in a controlled atmosphere on stainless steel foils of similar composition than the selected tubes. Tablets of Al and AlSi7 compositions cut from regular precursors were put on the foils, preventively cleaned and degreased, and then were slowly (0.6 °C/s) heated in the horizontal furnace up to 700 °C, in two different atmospheres, air or nitrogen.

Mechanical tests

Static uniaxial compression tests (loading rate = 1 mm/min) were conducted on the reinforced tubes by a 500 kN testing machine (Galdabini PM50). To evaluate the foam filling effect, axial compression tests were run on non-filled tubes and on the foamed cores as extracted from tubes after their expansion. In this case, to avoid interface reactions during foaming, the inner tube surface was preventively lubricated (by a graphitic powder).

Microstructural characterization

Metallographic specimens have been prepared from wetting tests and from tubes filled in air and in nitrogen. Interface microstructures were characterized by electron microscopy (SEM JEOL 5600) coupled with EDX microprobe analysis (OXFORD QUANTUM). Interface after the mechanical tests was also investigated and compared with results from the as-produced reinforced tubes.

Results and discussion

Direct foaming in tubes

A thermocouple inserted in one of the precursor's billets inside the tube allowed recording the temperature evolution during the foaming process (Fig. 2). The plateau region in each curve of Fig. 2 indicates the billet melting temperature which varies depending on the powder composition. In the Al curve, the plateau temperature, which is the aluminium melting point ($T = 660$ °C), is maintained until the precursor is totally melted, then the temperature suddenly raises. In the AlSi7 and AlSi3 plots, the first liquid formation matches with the AlSi12 eutectic temperature, at 577 °C, followed by a regular temperature increase as the liquid phase enriches in aluminium. In all cases, the successive steep temperature growth is an indication of the complete precursor melting and its transformation in a lump of foam. Actually, the precursor foaming begins since the first billet softening, although the foam reaches its maximum expansion when all the metal is liquefied

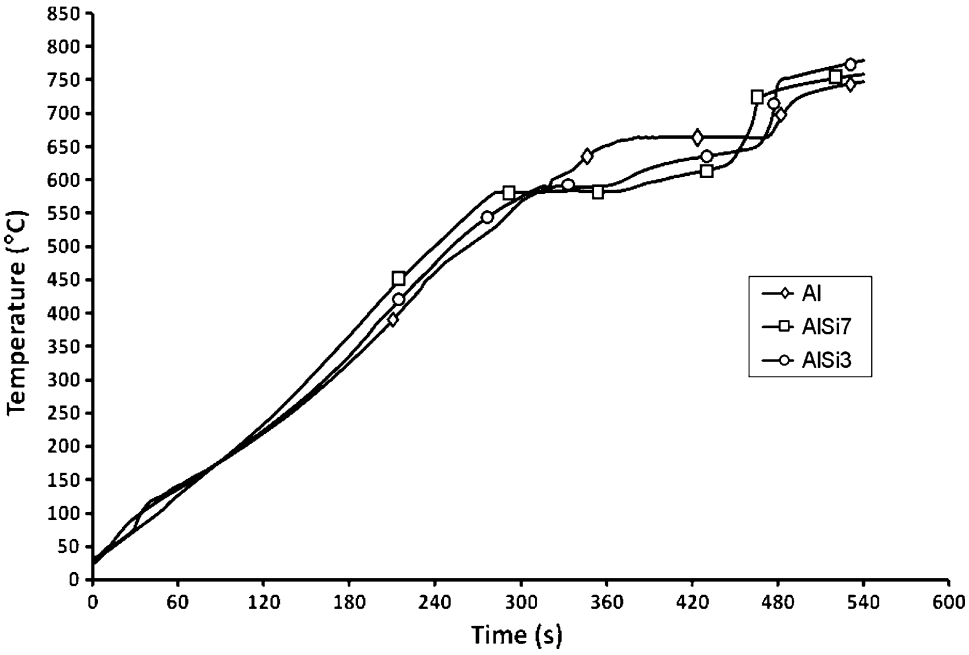


Fig. 2 Temperature evolution for the Al, AlSi7 and AlSi3 precursors during foaming inside tubes

[17, 18]. Foaming in a mould is a process difficult to control because of the several variables involved. Obvious differences between precursors, uneven temperature distribution during foaming, different thermal expansion between the core and the hollow element, intrinsic stochastic behavior of the foaming process itself, all those variables are responsible of the forming process scarce reproducibility. Some typical foaming defects observed in reinforced members are shown in Fig. 3. Pore coalescence (Fig. 3a), molten metal drainage and foam shrinkage (Fig. 3b) are frequently affecting the filling process. Usually, there are several concurrent causes for the observed defects, such as over-heating, or molten aluminium leakage through the lids and/or the effect of the different thermal expansion between aluminium and the tube alloy during cooling. Therefore, not only the foam formation but also the tube filling is important in order to get the expected improvements by the aluminium reinforcement.

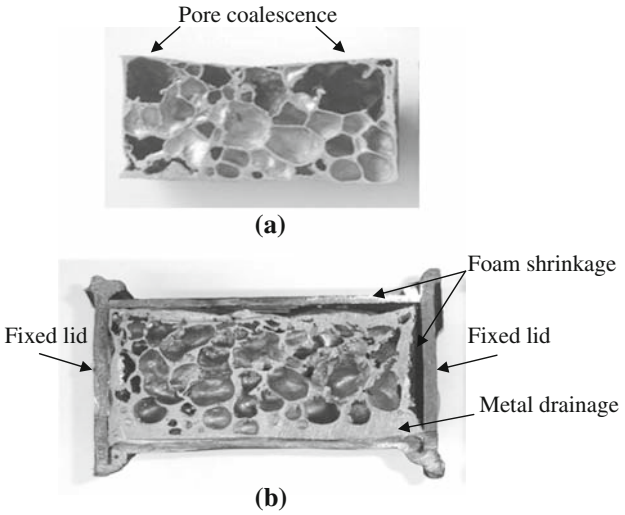


Fig. 3 Examples of filling defects: pore coalescence (**a**), foam shrinkage and drainage (**b**)

The dilatometric system outlined in Fig. 1 was used to monitor the foaming process inside the tube by recording the piston position so that the profile filling could be correlated to the foam expansion [18]. In Fig. 4, the billet temperature and the related piston position versus time during a tube filling experiment are depicted. After an initial negative displacement caused by the tube thermal dilatation, the foam expansion was recorded only when the precursors were almost completely molten. In fact, the piston reached its maximum positive displacement right after the steeply temperature increase recorded in the precursor curve, corresponding to the maximum foam expansion. Actually, the initial foaming fills the tube radially and the piston does not show any positive displacement. Once the pipe section is full, the foam expand longitudinally pushing the piston outward (Fig. 4).

Interface phenomena characterization

The occurrence of a metallurgical bonding at the foam–tube interface is related to the possibility of a chemical reaction between the melting aluminium and the shell walls. The chemical reaction can occur if liquid aluminium wets the metal case, but the pre-existing oxide film covering the precursors and the enhanced foam oxidation during the heat treatment in air do not allow any sort of chemical bond at the foam–tube interface [19, 20]. Moreover, in direct foaming, the contact time between the expanding foam and the tube surface is very short, making the interface reaction more difficult. However, in wetting

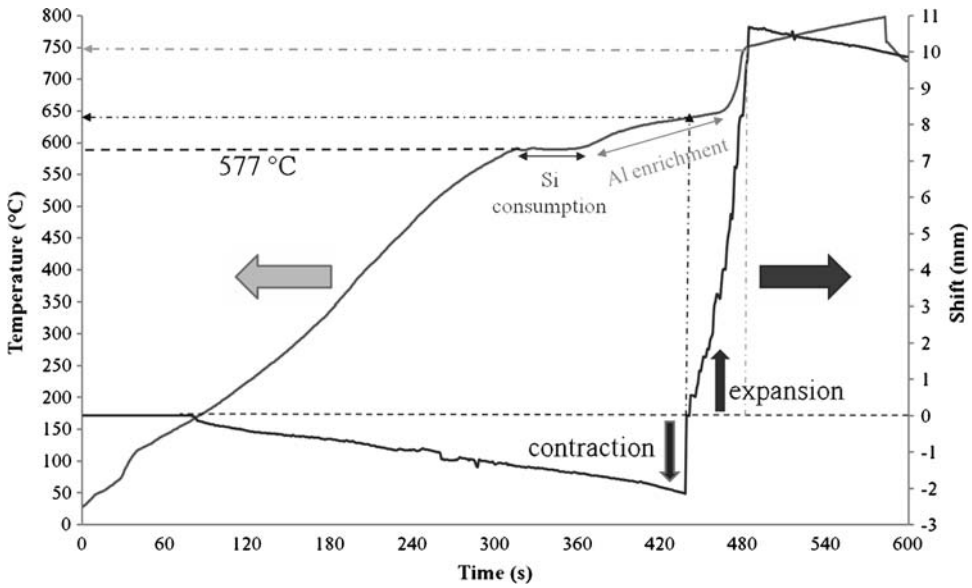


Fig. 4 Temperature and expansion curves of AlSi precursors during foaming

testes in nitrogen, it was observed that the foam bonded to stainless steel foils by an interface layer made of intermetallic phases. The transition zone was tongue-shaped on the aluminium side, whereas it was compact and uniform on the stainless steel side, for a total thickness of ≈30 μm (Fig. 5, the fracture crossing the section was produced during the sample polishing and preparation). The EDX element mapping and the electron probe microanalysis (Fig. 5) demonstrated that the layer nearest to the steel was mainly made of the intermetallic compound Fe_2Al_5 known as phase η, while the tongues extending on the aluminium side were made of the intermetallic compound $FeAl_3$ (phase θ) which was also observed distributed in the matrix (dendrites and grains) [21–24]. Nickel did not diffuse into the aluminium matrix, whereas chromium was observed close to the interface forming the intermetallic phase $CrAl_7$ (the coarse crystals in Fig. 5) and the ternary compound $Cr_{0.x}Fe_{0.y}Al_k$ (whisker-like grains) [24]. Microstructural investigation on tubes filled in air and nitrogen confirmed the wetting test results, i.e., the metallurgical bonding was observed mostly in non-oxidizing atmosphere. The

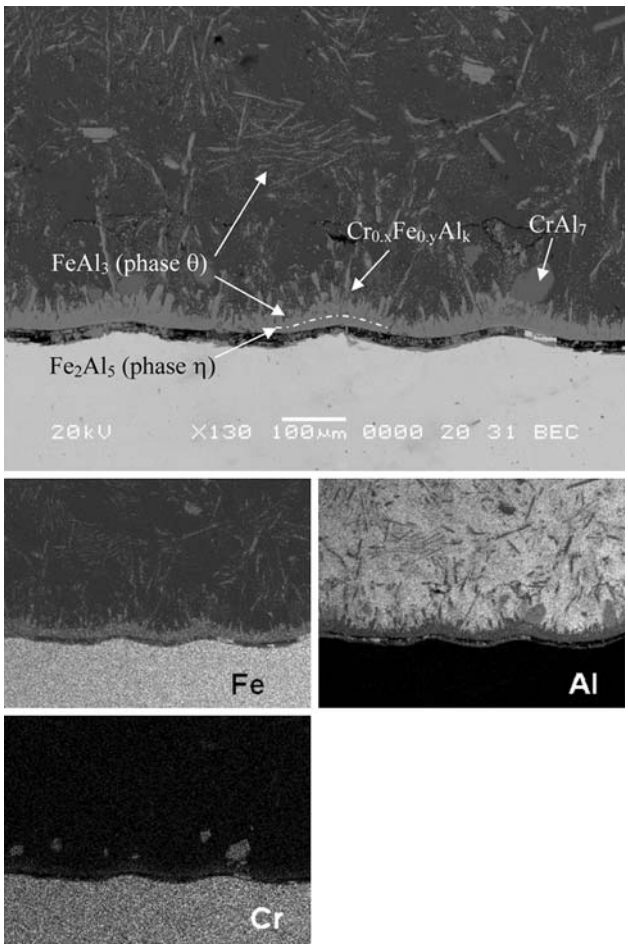


Fig. 5 Wetting test of Al foam on stainless steel foils in nitrogen

interface layer obtained in tubes filled in nitrogen formed almost continuously along the tube cross-sections and the morphology and composition of the transition zone was identical to that one observed in wetting tests, even if the thickness was lower, ranging between 7 and 10 μm (Fig. 6). By using AlSi7 precursors, a negative impact on wetting was expected to cause the Si presence [25]. However, a reaction at the foam–stainless steel interface was observed in wetting tests as well as in filled tubes when foaming was conducted in nitrogen. The bonding layer was quite difficult to analyze because of the formation of ternary intermetallic phases similar in composition (Fig. 7). Nearby the stainless steel surface, it was observed a compact layer mainly made of the $Fe_2(Al,Si)_5$ phase covered by coarse intermetallics grains of the Cr–Fe–Al system, while approaching the foam-side ternary intermetallics of the Fe–Al–Si system were observed (Fig. 7) [25]. Even in this case, the interface formation did not differ between wetting tests and filled tubes except for the layer thickness, which was always smaller in the tubes. Such a difference in thickness was attributed to the faster heating characterizing the foaming process than the wetting tests. Despite the short contact times of the filling process and the initial oxide film covering the precursors, the interface bonding in non-oxidizing conditions formed in the tubes too. During the foam filling, the friction against the tube walls of the expanding aluminium aided breaking the external oxide and some liquid non-oxidized aluminium emerged to the surface and reacted.

A significant improvement on the interface formation in oxidizing atmosphere was obtained by using the expansion set-up of Fig. 1. Loading the piston during foaming, the pore coalescence as well as the core cooling shrinkage were limited with a general enhancement in pore homogeneity and distribution (Fig. 8). Moreover, the metallurgical bonding was significantly extended to almost all the aluminium–steel interface, showing that constraining the foam expansion contributed to the steel surface wetting by molten aluminium (Fig. 8). This result confirmed that, even in oxidizing atmosphere, it was possible to improve the interface joining and the adhesion properties of the foamed reinforcement by increasing the attrition at the tube walls during foaming. From an operational point of view, the "constrained expansion" could represent an interesting alternative to the more difficult furnace atmosphere control in improving the bond between the foam and the hollow profile.

Mechanical testing

The specific high strength and stiffness which characterize the mechanical performance of metal foams have been largely investigated in very different conditions, from the

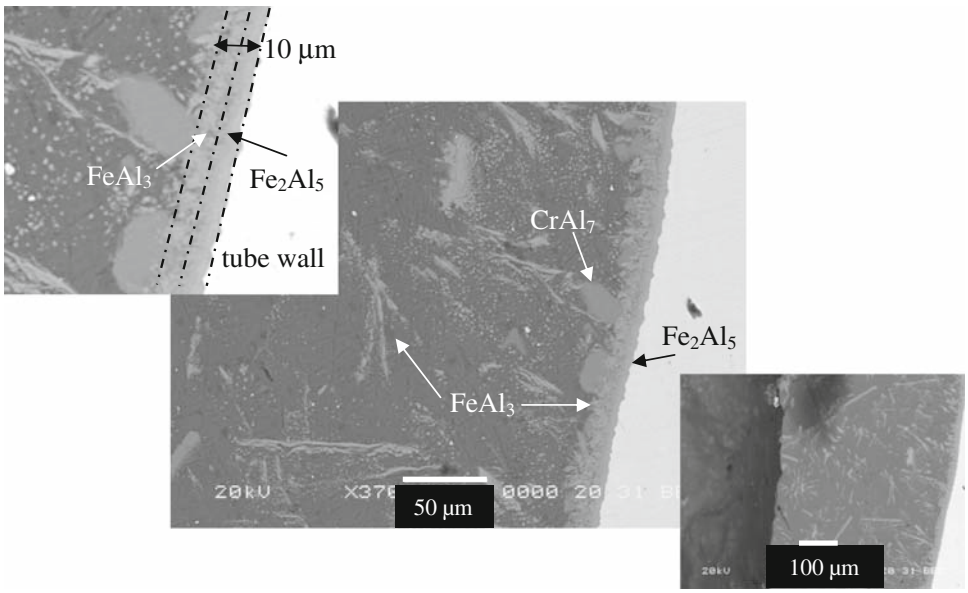
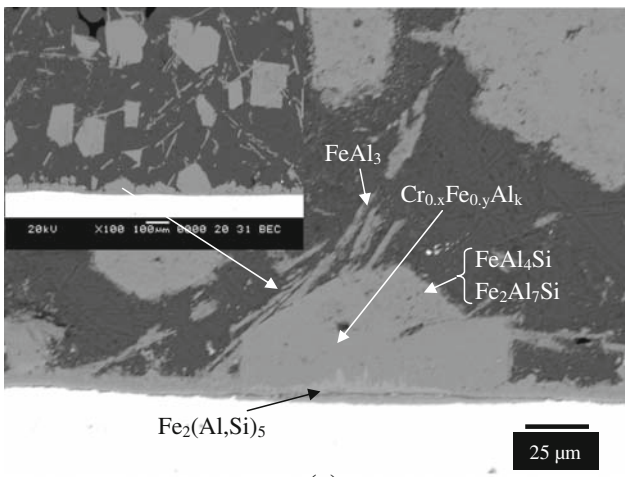
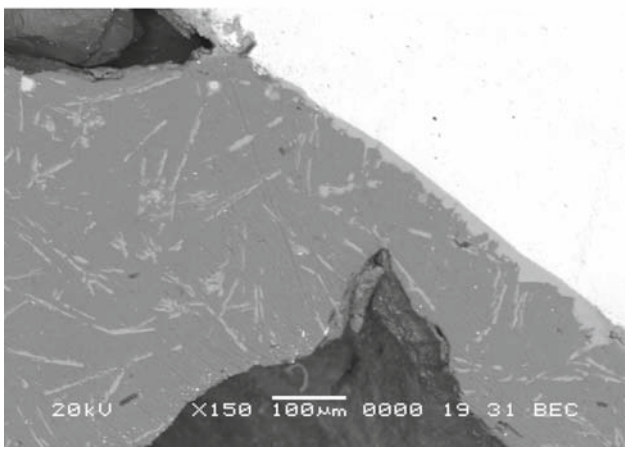


Fig. 6 Microstructure of the Al foam–tube (AISI 304) interface obtained in N_2



(a)



(b)

Fig. 7 Microstructure of the AlSi7 foam–stainless steel interface obtained in N_2. Wetting test (**a**) and tube cross-section (**b**)

nanoporous scale [26] to high-temperature applications [27, 28]. Figure 9 shows the static compression curves for the foamed core alone, the empty stainless steel tube, the algebraic sum of these two and, finally, for the reinforced structure. The cylindrical foamed core demonstrated the typical behavior of similar cellular structures with an extended plastic plateau and a compressive strength (the initial peak stress) of ~10 MPa, which is in good agreement with literature data [1, 29]. The empty stainless steel tubes showed a typical non-axisymmetric collapse mode named "diamond" [11] and not a significant deterioration of the mechanical properties was observed even after a heating treatment similar to the one used in the foaming process. The deformation mode as well as the number of lobs formed is dependent upon geometry and material properties. The compression response of the reinforced structure demonstrated a significant improvement in the absorbed energy, which was attributed to an "interaction effect" between the foamed core and the solid shell (Fig. 9). Several research papers on thin-walled columns filled with aluminium foams in static, quasi-static [4–6] and dynamic conditions [11–13] have demonstrated, indeed, that the high energy absorption is due to a change in the folding mechanism during the column buckling from the non-axisymmetric ("diamond" mode of the hollow tube) to the axisymmetric mode ("concertina" mode of the foam-filled tube) showing a shorter folding wavelength (Fig. 9). The increased numbers of tube folds and the multiaxial compression of the foam (instead of uniaxial as in the foam alone) justify for the higher energy absorbtion [11–14]. However, the efficiency of the energy absorption can be severely limited if no transverse strain occurs in the foam but the tube walls pull away from the core [13]. A poor

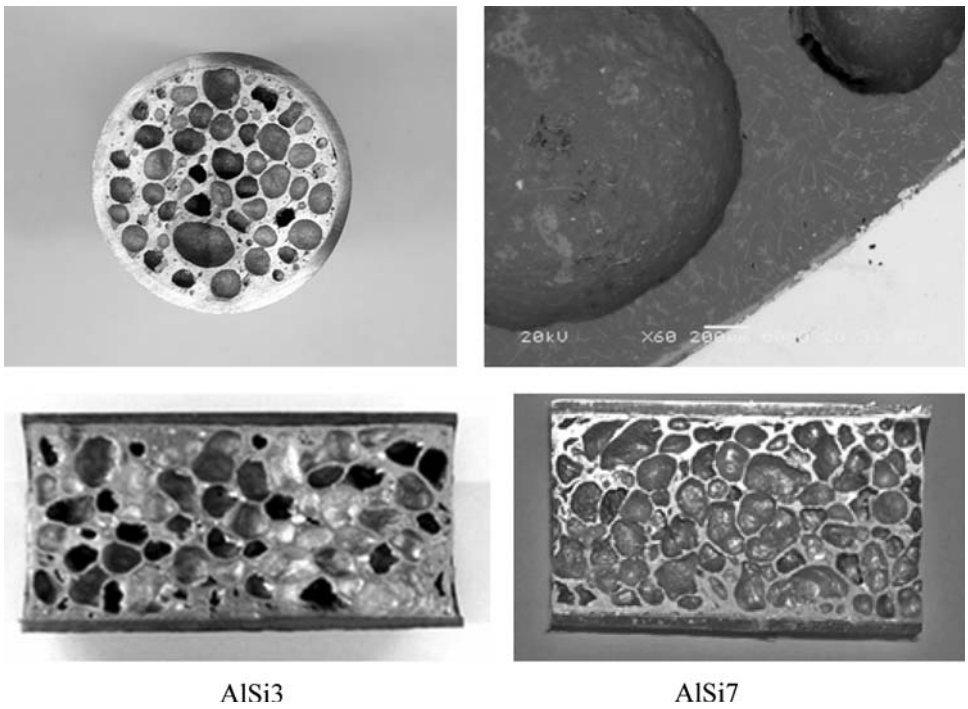


Fig. 8 Samples of tubes filled in air by "constrained expansion"

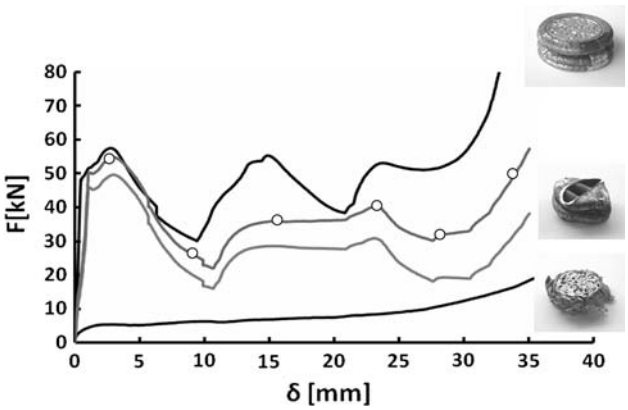


Fig. 9 Static uniaxial compression curves for the foamed core, the tube and the filled tube. (*Open circle*) Algebraic sum of the first two curves

bonding at the core–tube interface is, generally, the cause of the earlier detachment and the reduced transverse foam deformation. Figure 10a shows the longitudinal section of a compressed filled tube before the second fold was completed (the first from the top) and the third one (in the centre) was beginning to form. In this case, the tube was filled in air (without constraint) and the surface aluminium oxide prevented interface reactions. The foam did not penetrate in the tube folds because of the inefficient bonding that was not able to transfer tangential stresses to the core so that the foam deformed as it was not coerced by the tube, reducing the expected "interaction effect". When the interface formed, thanks to the non-oxidizing atmosphere or during the constrained expansion, the reinforcement intruded deeply into the tube foldings, absorbing a larger amount of deformation energy (Fig. 10). A confirmation of the strength of the interface bonding can be seen in Fig. 11, where SEM images of a collapsed tube fold are depicted. In Fig. 11a2 is evident a fracture line crossing the filler at ~ 50 μm from the interface which forms a chip of the same thickness facing the foam intrusion. Since the former investigations (see section "Interface phenomena characterization") have shown that in uncompressed samples the interface layer, made of phases η and θ, had a maximum thickness of ~ 10 μm (Figs. 6 and 7) while the observed fractured front propagated deeper in the filler, it is possible to argue that the interface bonding should have a mechanical strength almost comparable to the aluminium matrix, even if constituted of brittle phases [24]. These results confirm the value of a metallurgical bonding between the foamed aluminium and the steel tube and the relevance in producing an interface reaction during the production process.

Conclusion

The formation of a metallurgical bonding in reinforced stainless steel tubes produced by direct foaming was investigated. The tubes were filled in different conditions, in air, in nitrogen and in a "constrained expansion" condition. It was observed that the high-temperature oxidation of the foam surface was the main reason for an

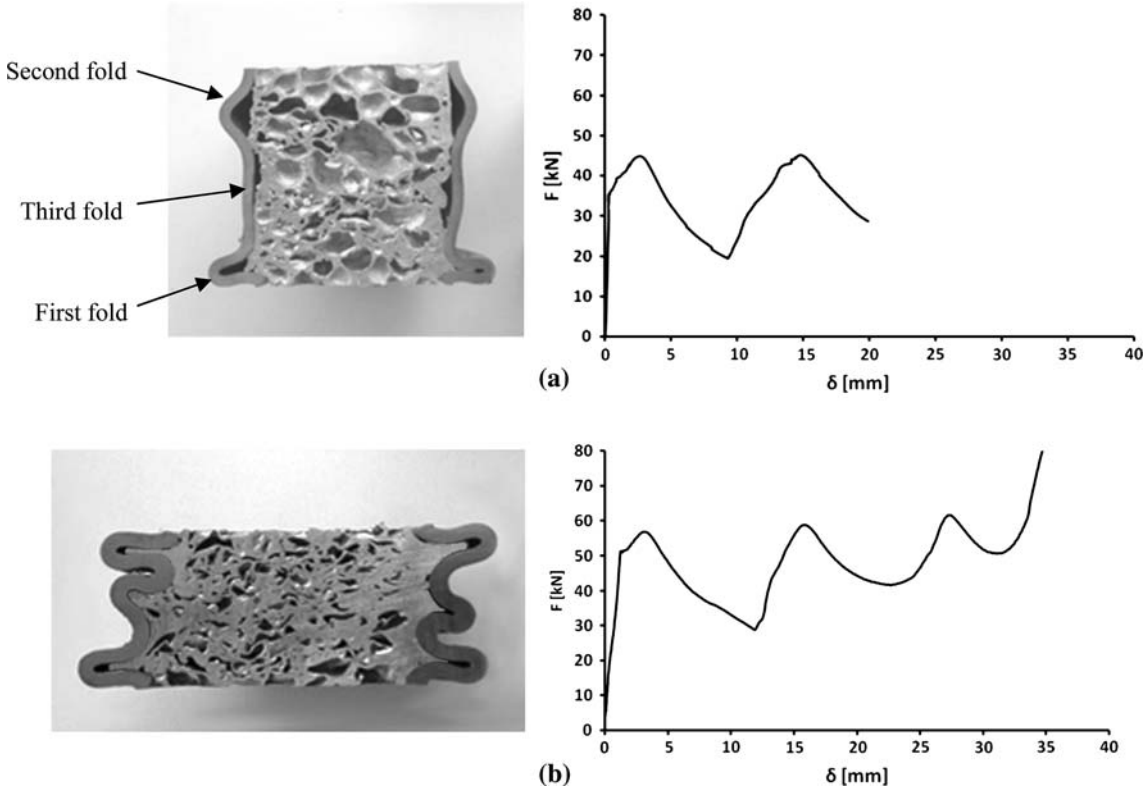
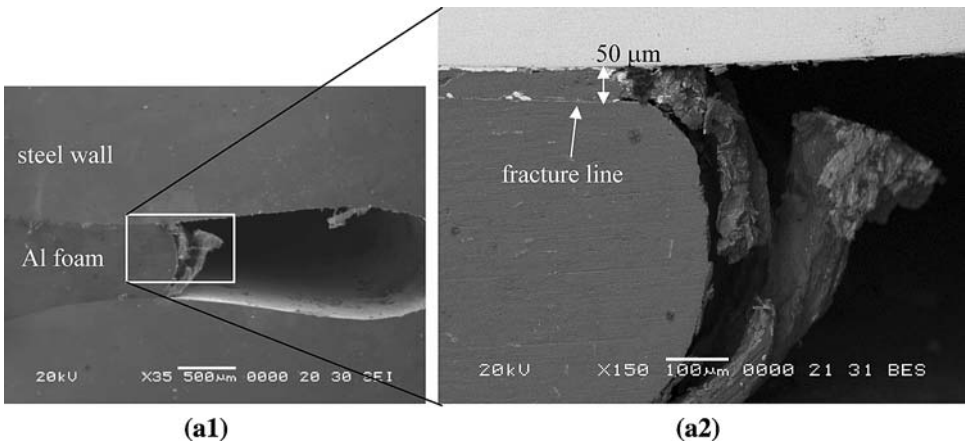


Fig. 10 Longitudinal sections of collapsed tubes. Sample of tube filled in air (**a**) and sample of tube filled by "constrained expansion" (**b**)

Fig. 11 SEM images of the aluminum foam intruded into the tube folds



inefficient interface bonding. However, a chemical reaction between the two components, the core and the shell, is possible, even during the short time requested for the foaming process, if the oxidation is limited. This can be obtained by operating in a controlled, non-oxidizing, atmosphere or by promoting the surface oxide breaking. The transition zone, when formed, was mainly made of intermetallics belonging to the Fe–Al system, whereas silicon (from the foam) and chromium (from the tube) showed lower reactivity. The interface bonding demonstrated an important role in the mechanical response of the reinforced member. The mechanical strength of the intermetallic layer resulted higher than expected significantly influencing the composite deformation mode. On the other hand, the lack of an interface bond prevented the foam from cooperating to the plastic deformation of the system. It can be concluded that in general a better control of the foaming process parameters in preparing reinforced structures can improve foam porosity and distribution as well as adhesion properties leading to an increased reproducibility and reliability of metal-foamed components.

References

1. Ashby MF, Evans A, Fleck NA, Gibson LJ, Hutchinson JW, Wadley HNG (2000) Metal foams: a design guide. Butterworth-Heinemann, Boston
2. Ito K, Kobayashi H (2006) Adv Eng Mater 8(9):828
3. Stöbener K, Rausch G (2009) J Mater Sci 44:1506. doi:10.1007/s10853-008-2786-8
4. Haberling C, Haldenwanger HG (1999) In: Banhart J, Ashby MF, Fleck NA (eds) Metal foams and porous metal structures. MIT Verlag, Bremen
5. Langseth M, Hopperstad OS, Hanssen AG (1998) Thin-Walled Struct 32:127
6. Santosa S, Wierzbicki T (1998) Comput Struct 68:343
7. Peroni L, Avalle M, Peroni M (2008) Int J Impact Eng 35:644
8. Idris MI, Vodenitcharova T, Hoffman M (2009) Mater Sci Eng A 517:37
9. Guden M, Yüksel S, Taşdemirci A, Tanoğlu M (2007) Compos Struct 81:480
10. Korner C, Heinrich F, Singer RF (2002) In: Degischer HP, Kriszt B (eds) Handbook of cellular metals: production, processing, applications. Wiley-VCH Verlag GmbH, Weinheim
11. Hanssen AG, Langseth M, Hopperstad OS (2000) Int J Impact Eng 24:475
12. Hall IW, Ebil O, Guden M, Yu CJ (2001) J Mater Sci 36:5853. doi:10.1023/A:1012916408297
13. Hall IW, Guden M, Claar TD (2002) Scr Mater 46:513
14. Grandinger R, Seitzberger M, Rammerstorfer FG, Degischer HP, Blaimschein M, Walch Ch (1999) In: Banhart J, Ashby MF, Fleck NA (eds) Metal foams and porous metal structures. MIT Verlag, Bremen
15. Seitzberger M, Rammerstorfer FG, Grandinger R, Degischer HP, Blaimschein M, Walch Ch (2000) Int J Solids Struct 37:4125
16. Simancik F, Schoerghuber F (1998) In: Schwrtz DS, Shih DS, Evans AG, Wadley HNG (eds) Material research society, vol 521. Warrendale, Pennsylvania
17. Duarte I, Banhart J (2000) Acta Mater 48:2349
18. Karsu ND, Yüksel S, Güden M (2009) J Mater Sci 44:1494. doi:10.1007/s10853-008-3039-6
19. Kaptay G (1991) Materials science forum, vol 77. Trans. Tech Publications, Switzerland
20. Chatillon C, Coudurier L, Eustathopoulos N (1997) Materials science forum, vol 251–254, Trans. Tech Publications, Switzerland
21. Vendra LJ, Rabiei A (2007) Mater Sci Eng A 465:59
22. Rabiei A, Vendra LJ (2009) Mater Lett 63:533
23. Barmak K, Dybkov VI (2003) J Mater Sci 38:3249. doi:10.1023/A:1025129803413
24. Barmak K, Dybkov VI (2004) J Mater Sci 39:4219. doi:10.1023/B:JMSC.0000033402.37206.27
25. Fragner W, Zberg B, Sonnleitner R, Uggowitzer PJ, Loffler JF (2006) Materials science forum, vol 519–521. Trans. Tech Publications, Switzerland
26. Xia R, Xu C, Wu W, Li X, Feng Xi-Q, Ding Y (2009) J Mater Sci 44:4728. doi:10.1007/s10853-009-3731-1
27. Azarmi F, Coyle TW, Mostaghimi J (2009) J Mater Sci 44:2836. doi:10.1007/s10853-009-3375-1
28. Cluff D, Esmaeili S (2008) J Mater Sci 43:1121. doi:10.1007/s10853-007-2271-9
29. Sugimura Y, Meyer J, He MY, Bart-Smith H, Grenstedt J, Evans AG (1997) Acta Mater 45:5245