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W. SZYMAŃSKI\*<sup>#</sup>**PROPERTIES OF „IN SITU” 7475 ALUMINIUM ALLOY MATRIX COMPOSITES REINFORCED  $Al_3Ti$  INTERMETALLIC COMPOUND, EXTRUDED IN SEMI-SOLID STATE**

Studies were conducted to improve the mechanical properties of composites based on 7475 aluminium alloy reinforced with  $Al_3Ti$  particles fabricated by the “in situ” process.

The first step involved “dissolving” of titanium in the liquid aluminium alloy and fabricating in this way composite materials with different content of the reinforcing phase (15-45wt%). A relationship between the composite hardness and content of the reinforcing phase was confirmed.

The second step involved the improvement of cohesion between the reinforcing particles and composite matrix. By extrusion of samples in semi-solid state, an average increase in hardness by 15-20% relative to the unextruded composite was obtained.

In the third step, the fabricated composite was subjected to a heat treatment corresponding to the state T6 in 7475 alloy, which raised the hardness by about 30%. Structure examinations carried out by means of optical microscopy and scanning electron microscopies as well as the results of hardness measurements were described. They enabled estimating the effect of the content of produced  $Al_3Ti$  particles, and of the extrusion process in semi-solid state and heat treatment parameters on the composite properties. In compression test, the yield strength and compressive strength of the heat-treated composites were determined.

*Keywords:* Al-matrix composites, in situ composites, extrusion in semi solid state, heat treatment,

**1. Introduction**

Composites based on aluminium and its alloys have already been used for several decades [1]. The process of their fabrication assumes the addition of 5-50% of reinforcing particles introduced into the composite volume. In this respect, a very serious challenge to modern engineers is how to ensure an effective bond between the composite matrix and the reinforcing phase, as only in this way it can be expected that both phases of the material will correctly perform their task.

The “in situ” method is one of the most promising techniques in the fabrication of composites and surface engineering of the bonded components.

Relevant data on the “in situ” composites are provided in a monograph written by S.C. Tjong and Z.Y. Ma [2] and X.Wang, A.Jha and R.Brydson [3]. They believe that the “in situ” composites are characterized by a homogeneous distribution of reinforcement and good mechanical properties.

Efforts continue to seek opportunities to improve the properties of composites based on Al-Ti. One of such methods might be the use of a plastic forming process.

Aluminium composites produced by liquid phase extrusion have been studied in [4,5]. It was found that composite samples subjected to radial extrusion do not crack despite the use of high strain, thus demonstrating substantial plasticity. The use of an extrusion process in which there is

the most favourable hydrostatic stress state leads to a good consolidation of the composite components.

One of the most important issues in the fabrication of composites is how to maintain a good consistency between the joined components. Produced by the dissolution of Ti in liquid Al or its alloy, the “in situ” Al- $Al_3Ti$  composites, although improving greatly their properties with the increasing content of a reinforcing phase, show the presence of fine pores and micropores [6]. Pores in the composite structure are the result of differences in the thermal expansion coefficient of Al (or Al alloy) and  $Al_3Ti$  intermetallic phase, resulting in the loss of cohesion during composite cooling. The occurrence of microporosity in the composites is related with changes in the volume of the aluminium-titanium system due to the formation of an  $Al_3Ti$  reinforcing phase (Kirkendal effect) [7]. This results in the formation of discontinuities at the matrix - reinforcing phase interface and leads to unfavourable structural changes. An increase in porosity was stated with the increasing content of  $Al_3Ti$  phase. It has been concluded that to eliminate this defect further processing of the composite might prove useful and reduce the formation of discontinuities at the composite interfaces. To this end it was decided to apply the plastic working process in semi-solid state as a means to improve the composite structure.

Analysis of the literature data has showed that one of the most efficient processes improving phase consistency is the extrusion in semi-solid state. The use of aluminium

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alloys as a composite matrix has created not only the possibility to obtain higher mechanical properties, but also better balancing of the liquidus-solidus temperature and achieving the desired effect of interaction between the solid and liquid phase, and in the case of the latter one also high degree of penetration into every pore and discontinuity present in the sample.

The study describes the research on the improvement of mechanical properties of the “in situ” fabricated 7475 aluminium alloy matrix composite reinforced with  $\text{Al}_3\text{Ti}$  particles. The improvement was due to a better cohesion between the reinforcement particles and matrix obtained by the application of semi-solid extrusion with the following heat treatment.

## 2. Research methodology

To produce the composite, the following materials were used:

- aluminium of 99.99% purity,
- BT1-0 titanium strip (0.25 Fe, 0.10 Si, 0.07 C, 0.12 O, 0.04 N, 0.010 H, 0.30 other elements) with a thickness of 0.25 mm and a width of 20 mm,
- 7475 alloy of the composition shown in Table 1.

TABLE 1

The composition of 7475 alloy used in this study [wt%], rest – Al

| Element       | Fe   | Si   | Cu  | Zn   | Ti    | Mn    | Mg  | Ni    | Zr   |
|---------------|------|------|-----|------|-------|-------|-----|-------|------|
| Content [wt%] | 0.09 | 0.07 | 1.5 | 5.55 | 0.016 | 0.006 | 2.2 | 0.037 | 0.27 |

To produce the  $\text{Al}_3\text{Ti}$  intermetallic compound, which was used as a composite reinforcement, a method involving the dissolution of titanium strip in molten aluminium or its alloys was used.

The applied method of the in-situ manufacture of composites involves the synthesis of reinforcing phase ( $\text{Al}_3\text{Ti}$ ) formed directly in the liquid metal [6]. Titanium strip placed in special crucible was poured with liquid aluminium or aluminium alloy. The material was next annealed at a temperature of about 800°C. As a result of titanium entering into reaction with the liquid aluminium, equiaxed particles of the intermetallic phase of  $\text{Al}_3\text{Ti}$  were formed (Fig. 1).

To obtain the composite material with a predetermined volume fraction of the  $\text{Al}_3\text{Ti}$  phase, it was necessary to account in the calculations for the fact that during the formation of this phase, the matrix would lose some aluminium, transferred to the newly formed  $\text{Al}_3\text{Ti}$  phase. Therefore, an extra amount of aluminium (twice the weight of the added Ti) was added to maintain the assumed composition of the matrix.

The components were chosen in such a way as to obtain a composite containing 10, 20, 30 and 40 wt%  $\text{Al}_3\text{Ti}$ .

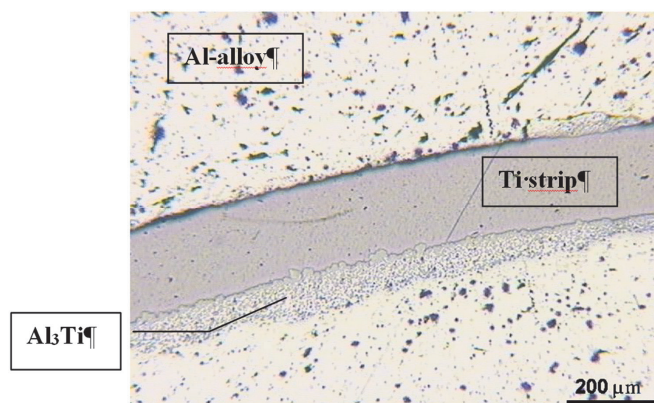


Fig. 1 Onset of the Ti strip dissolution in liquid aluminium alloy and the first stage of the  $\text{Al}_3\text{Ti}$  phase formation

The composites with varying volume fraction of the  $\text{Al}_3\text{Ti}$  phase were tested for the structure condition and properties.

In the second stage of research, the composites were extruded in semi-solid state, and the obtained samples were again subjected to structure examinations and testing of properties.

The extrusion of composites was performed in a device designed especially for the purpose of this research. The device was adapted for use with a vertical hydraulic press of 500 kN capacity.

Trials of direct extrusion of the manufactured composites were carried out under isothermal conditions, created by heating the sample material together with the extrusion device, which was next mounted in a press. To this end, both device and sample material were placed in a sillite furnace and held there for 2 hours, to be transferred next directly from the furnace to a hydraulic press, where the process of direct extrusion was conducted at a low speed. Before transfer to the furnace for heating, both sample material and tool were covered with molybdenum grease containing  $\text{MoS}_2$ , which retains good lubricating properties even at high temperatures. A large mass of the device, small dimensions of the sample, and short time of both installation in the press and extrusion ensured maintenance of the required temperature.

The feedstock in the form of billets of 30 mm diameter was extruded into rods with a diameter of 5.5 mm, i.e. applying the extrusion ratio  $\lambda \approx 30$ . The optimum material flow in the deformation zone was ensured by the use of conical die with the face inclined at an angle of 60° to the direction of extrusion. During the extrusion of composites based on aluminium alloys heated up to 640°C, in some cases an outflow of the liquid phase was observed in the initial stage of the process. To avoid this defect, the preheating temperature was reduced to 630°C, and in some cases even to 620°C. After the drop of temperature, this effect was observed no longer.

The heat treatment of composite after extrusion was carried out according to the following regime: solutioning from 470°C followed by aging at 120°C for 24 hours.

The structure of the samples was examined under an “Olympus GX71” optical microscope and “Philips XL30” scanning electron microscope. Computer analysis of the geometric parameters of structure constituents (volume and size of the  $\text{Al}_3\text{Ti}$  phase particles) was carried out by the “APHELION” programme. Hardness tests were performed



with a “HPO” hardness tester; the yield strength was determined in a compression test made on an “Instron 5582” machine.

### 3. Test results

The process of titanium dissolution and  $\text{Al}_3\text{Ti}$  phase synthesis in liquid aluminium alloy gave billets of the structure shown in Figures 2 and 3.

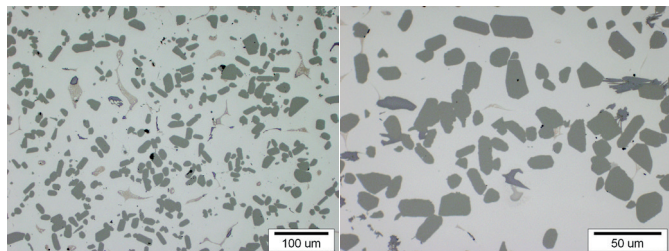


Fig. 2. Examples of the composite billet microstructure

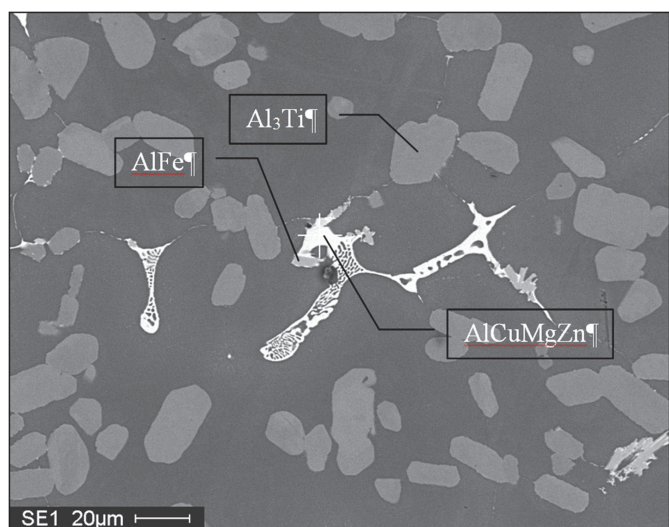


Fig. 3. Microstructure of the cast composite with chemical analysis of the  $\text{Al}_3\text{Ti}$  reinforcing phase precipitates

Volume fractions of the  $\text{Al}_3\text{Ti}$  phase calculated by the technique of computer-aided microstructural image analysis are shown in Table 2.

TABLE 2

Volume fractions of the  $\text{Al}_3\text{Ti}$  intermetallic phase calculated for composite billets

| Theoretical [wt%] | 10   | 20   | 30   | 40   |
|-------------------|------|------|------|------|
| Calculated [vol%] | 15,2 | 22,5 | 27,6 | 42,6 |
| St. dev.          | 4    | 3    | 2    | 8    |

In contrast, the average size of the  $\text{Al}_3\text{Ti}$  particles (also calculated by the technique of computer-aided microstructural image analysis) ranged from about  $15\mu\text{m}$  to about  $25\mu\text{m}$  and was independent of the reinforcement content in the composite.

Examinations microstructure confirmed the presence of pores in the non-extruded composites (Fig. 4a); in the extruded composites pores were absent (Fig.4b).

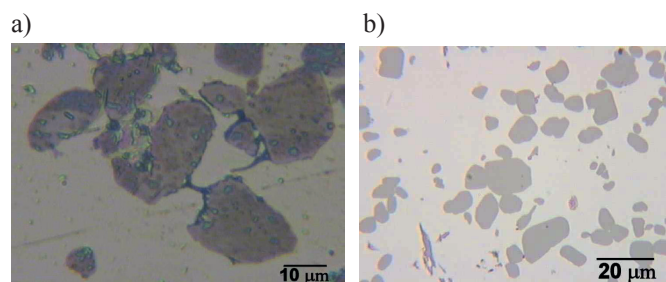


Fig. 4 Microstructure of the composite before (a) and after (b) extrusion

Differences between the composite non-extruded and extruded were even more pronounced in the microstructure near the hardness tester indentation. In the composite before extrusion, the deformation of material caused by the hardness tester indenter resulted in the formation of clear cracks at the interface (Fig. 5), while in the composite extruded in semi-solid state, the deformed material was free from cracks at the interface, and the slip lines extending from the matrix to the reinforcement (Fig. 6) indicated compact structure of the material. A decrease in the number of pores, determined by the analysis of microstructure images of the material extruded and non-extruded, was also observed (Fig. 7a).

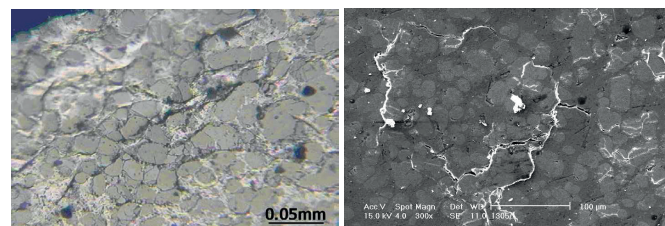


Fig. 5. Non-extruded composite microstructure in the vicinity of hardness tester indentation

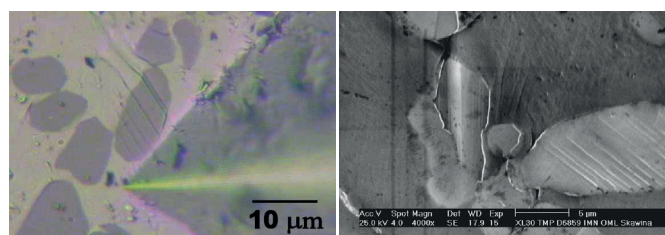


Fig. 6. Extruded composite microstructure in the vicinity of hardness tester indentation

The results of hardness measurements (Fig. 7b) confirmed the composite hardness increase with an increase of the reinforcing phase content and improved properties after the extrusion and heat treatment.

Small dimensions of the available samples were the reason why for testing of the composite mechanical properties the compression test was chosen.

A diagram in Figure 8a shows the yield strength and breaking stress of the tested 7475 alloy matrix composites. The breaking stress is the maximum stress at which the failure of the sample occurs (Fig.8b).

Dividing line after the destruction samples both on the matrix and the reinforcement also well testifies to the cohesion of the material (Fig.9).

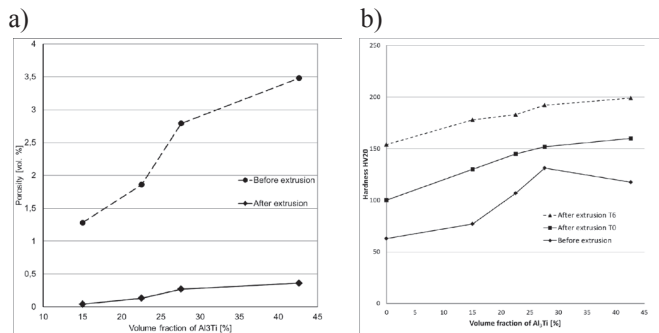


Fig. 7. Hardness (a) and porosity (b) changing in the composite before and after extrusion in relation to the reinforcement content

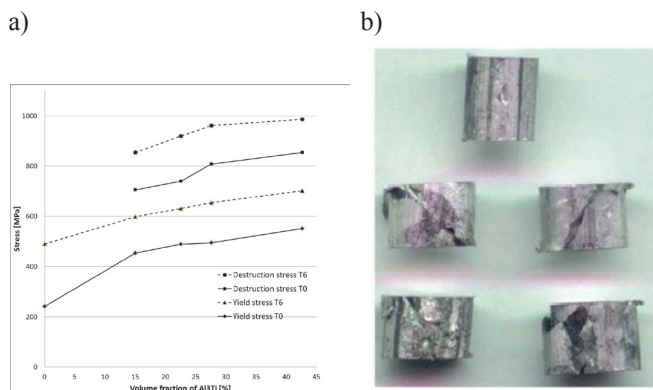


Fig. 8. Changes in the value of  $R_{p0.2}$  and breaking stress as a function of the reinforcement content in the composite extruded (a) and (b) examples of compressed samples (the top one - before testing)

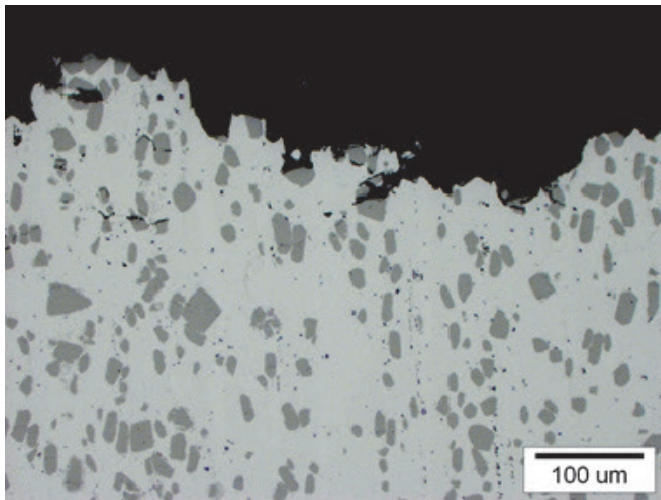


Fig. 9. Composite structure around cracks

#### 4. Discussion of results

Strong bond between the Al<sub>3</sub>Ti phase and matrix was evidenced by hardness measurements, which clearly indicated an increase of hardness in the extruded samples as compared with the samples before extrusion. Studies of the composites extruded in semi-solid state clearly show that the compressive stresses applied during high-temperature bonding of phases remove the discontinuities and promote their fusion. They also remove the internal stresses, which disappear in the presence of the liquid phase, thus promoting further bonding

of the composite components. This confirms the phenomenon described in the works of other authors [2, 3, 8, and 9]. A well noticeable structural effect after the extrusion in semi-solid state is the presence of slip lines extending through the matrix and reinforcing phase, and thus indicating a fast bond between the phases.

Another tested parameter was the content of pores in samples before and after the extrusion. Considerable reduction in the amount of pores and voids in the extruded samples was observed. The reduced presence of discontinuities was confirmed by hardness measurements taken before and after the extrusion. Furthermore, structure examinations by light microscopy and scanning electron microscopy showed a marked decrease in the areas containing such discontinuities. The reduced level of porosity was also confirmed by studies of micropores observed in the structure of the samples.

Beneficial effect of the semi-solid composite extrusion was most evidently manifested in the increased hardness and yield strength of this material when processed by extrusion. The yield strength of the 7475 alloy (241 MPa without reinforcement) increased to approx. 550 MPa after the introduction of the reinforcing phase. Heat treatment resulted in further increase of the composite properties, raising the yield strength by about 150 MPa and hardness by about 50HV

#### 5. Conclusions

1. The, extruded in semi-solid state, 7475 alloy-Al<sub>3</sub>Ti composite is characterised by high mechanical properties and good consistency of phase components,
2. Structure examinations showed significant improvement in the consistency of the composite components after the extrusion process in semi-solid state,
3. A significant increase was obtained in both the yield strength and compressive strength of the composites compared with the yield strength and compressive strength of the alloy matrix in base state.

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