Nanomechanical behaviour of Al-Ti layered composites produced by accumulative roll bonding

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Abstract. In this study Ti-foils were roll bonded together with commercially pure aluminium AA1050. The laminates were produced by using two 1 mm thick AA1050 sheets at the outer side of the stack combined with 100 µm thick Ti-foils as an intermediate layer for each accumulative roll bonding process. The samples were rolled up to 4 ARB cycles. Subsequently the sheets were post-process heat treated at 180°C, 400°C or 600°C, respectively, for 24 hours. The local mechanical behaviour of the Al/Ti intermetallic interfaces have been investigated using nanoindentation experiments. A strong dependence between annealing-temperature, -time and deformation grade is detected. While a heat treatment at 180°C only leads to a weak bonding between Al and Ti with a preservation of the UFG structure, temperatures up to 600°C are causing a complete recrystallisation of the microstructure and formation of diffusion layers with different Al and Ti concentrations.

1. Introduction
Over the last decades, nanocrystalline and ultrafine-grained (UFG) materials with grain sizes of less than 1 µm have gained significant attention. Especially their mechanical properties in terms of strength and ductility show superior behaviour compared to conventionally grained materials [1,2].

Besides the production of UFG sheet materials the accumulative roll bonding (ARB)-process [3-5] provides the very promising possibility to produce composite materials with a layered structure or even laminates. Thus, it is possible to insert different types of particles, fibres and foils to create specific laminates, multistacks and graded materials with tailored properties [6].

Especially the intermetallic compounds of the Al/Ti system have a great potential for industrial applications. In the past, investigations on the diffusion reaction between pure Al and pure Ti were accomplished by Wei et al. [7].

In the current work Ti-Al laminates were produced by ARB and the local behaviour of the Al/Ti-interfaces were systematically studied with nanoindentation.

2. Experimental procedure

2.1. Materials
Aluminium of commercial purity Al 99.5 (equivalent to AA1050A) and Titanium of commercial purity Ti (grade 1) were used. The chemical composition of both materials is listed in Table 1. Prior to
the accumulative roll bonding process, the aluminium was solution heat treated at 500°C for 1 h and subsequently quenched in water, while titanium was annealed at 700°C for 15 min and air cooled.

Table 1. Chemical composition of commercially pure aluminium AA1050A and pure Ti (grade 1).

|          | wt % | Si  | Fe  | Cu  | Mn  | Mg  | Zn  | Ti  | others | Al |
|----------|------|-----|-----|-----|-----|-----|-----|-----|--------|----|
| AA1050A  |      | 0.25| 0.40| 0.05| 0.05| 0.05| 0.07| 0.05| 0.03    |    |
| Ti (grade 1) |      | 0.12| 0.15| 0.05| 0.06| 0.013| 0.013| 0.40| balance |

2.2. Accumulative roll bonding process (ARB)
The size of the aluminium sheets prior to the roll bonding process were 100 mm x 300 mm x 1 mm, the titanium foils were 20 mm x 300 mm x 100 µm in size. The surfaces of both materials were wire brushed in order to remove oxide layers. The aluminium sheets were stacked on top of each other with the Ti-foil as an interlayer between the two aluminium matrix sheets. Afterwards the stacks were rolled together without lubrication to a 50% thickness reduction using a four high rolling mill (Carl Zeiss, Mühlacker). The bonded, laminated metal sheets were halved, folded and once again wire brushed before the next cycle. The sheets were roll bonded up to 4 cycles each time with two interlayer of Ti-foil. The principle is shown in Figure 1. Finally, the produced Al/Ti laminate structures were post-process heat-treated at 180°C, 400°C and 600°C.

2.3. Investigation of microstructure and mechanical properties
All specimens were prepared for metallographic examination by sectioning transverse and longitudinal to the rolling direction. The specimens were mounted in acrylic resin Technovit 2000 LC, ground, polished with diamond down to 1 µm and electro polished.

Light microscopy, scanning electron microscopy and energy dispersive spectrometry were used to investigate the microstructure and the occurred phases and compounds.

For investigating the local mechanical behavior of the Al/Ti interface as well as the occurred phases, nanoindentation experiments were carried out [8]. The experiments have been performed on a G200 Agilent Nano Indenter with a Berkovich diamond tip and a load controlled method using 20 mN as the maximum load. Arrays of 3 x 12 indents were directly positioned over the Al/Ti interfaces. Neighboring indents were separated by a distance of 20 µm to avoid an influence of the affected plastic zone around an indent.

3. Results and Discussion

3.1. Structure
Figure 2 shows the morphology of Ti fragments in the Al/Ti laminates after 4 cycles of ARB. It is obvious that more fragmentation occurs in the longitudinal section than in the transversal section. For
both sections the Ti fragments are homogenously spread in the Al matrix, but depending on the orientation of the cross section the number and size are different. The longitudinal section shows a multiplicity of short, small Ti fragments, whereas in the transversal section very long, thin Ti parts are dominating. Especially in the last bonding layer (middle layer), the transversal Ti fragment is very long and not cracked.

Figure 2. Light microscopic images of the morphology of Ti fragments in Al/Ti laminates after 4 cycles ARB in transversal (a) and longitudinal (b) section

In order to qualify optically the Al/Ti interface behavior, three samples were prepared by using the Focused Ion Beam (FIB) technique. Prior to the preparation, the samples were heat treated at 180°C, 400°C or 600°C, respectively for 24 hours. The prepared cross sections are showing nicely that 180°C and 400°C are not leading to any diffusion reaction between Al and Ti. Only after a 600°C heat treatment for 24 hours an interdiffusion zone occurs consisting of tiny phases, which are embedded in the Al matrix. The newly formed phase was detected to be TiAl3 by EDS analysis (Figure 5).

Figure 3. Scanning electron microscopic images of Al/Ti interfaces after various heat treatments at (a) 180°C; (b) 400°C; (c) 600°C prepared by Focused Ion Beam cuts

3.2. Mechanical properties

The different heat treatments are leading to a hardness decrease in the Al matrix, as shown in Figure 4. The local hardness of the ultrafine-grained Al-matrix before annealing averages at 0.6 GPa. First recovery processes and little grain growth occurs during low annealing temperatures of 180°C. The local hardness decreases to 0.51 GPa with a relatively high standard deviation. This argues for a less inhomogeneous grain structure, while the UFG structure is mostly preserved. Further increasing of the annealing temperature leads to a continuous decrease in the hardness to 0.42 GPa.

Figure 4. Hardness of Al with increasing annealing temperature (each after 24 hours heat treatment).
The results of the local hardness measurements obtained by nanoindentation experiments through a cross section of the Al/Ti laminate are shown in Figure 5. The compound material was post-process heat treated at 600°C for 24 hours, which led to a formation of an intermetallic TiAl3 phase determined by EDS. Figure 5 shows clearly the dependence between local hardness and chemical composition. The local hardness of the averages at 0.4 GPa for pure Al and respectively 2.1 GPa for pure Ti.

![Figure 5. Local hardness evolution and EDS linescan across the Al/Ti multimaterial stack after annealing at 600°C](image)

Comparing the results, it can be shown that the new formed intermetallic phase has a significant higher hardness as pure Al and Ti. The absolute value averages between 4 GPa up to 7 GPa. The value distribution can be explained with the inhomogeneous structures of the formed two phase regions. During further nanoindentation experiments in compact TiAl3, hardness values of almost 9 GPa were observed.

4. Conclusion

In the present study Ti-foils were rolled in pure Al sheets by using the ARB process. The foils broke during the deformation process and were homogenously spread in the Al-matrix. The distribution of those Ti fragments differs between longitudinal and transversal section.

Annealing processes at 180° and 400° led to a weak bonding with plenty of surface defects, while a heat treatment at 600°C led to a formation of an intermetallic TiAl3 phase. Local mechanical properties could be successfully investigated by carrying out nanoindentation experiments.

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