FRICTION STIR WELDS OF Al ALLOY-Cu: AN INVESTIGATION ON EFFECT OF PLUNGE DEPTH

In the present study, butt joints of aluminum (Al) 8011-H18 and pure copper (Cu) were produced by friction stir welding (FSW) and the effect of plunge depth on surface morphology, microstructure and mechanical properties were investigated. The welds were produced by varying the plunge depth in a range from 0.1 mm to 0.25 mm. The defect-free joints were obtained when the Cu plate was fixed at the advancing side. It was found that less plunging depth gives better tensile properties compare to higher plunging depth because at higher plunging depth local thinning occurs at the welded region. Good tensile properties were achieved at plunge depth of 0.2 mm and the tensile strength was found to be higher than the strength of the Al (weaker of the two base metals). Microstructure study revealed that the metal close to copper side in the Nugget Zone (NZ) possessed lamellar alternating structure. However, mixed structure of Cu and Al existed in the aluminum side of NZ. Higher microhardness values were witnessed at the joint interfaces resulting from plastic deformation and the presence of intermetallics.

1. Introduction

Copper has excellent ductility, corrosion resistance, thermal and electrical conductivity, and has been widely used to produce engineering parts such as electrical component, switchgear and radiator etc. [1]. Aluminum and its alloys are lighter in weight, having high strength and can resist corrosion. It can be easily fabricated and thus these properties make it desirable for a wide variety of applications. These days, the bimetallic joints, particularly Al to Cu, are progressively being used in a number of electrical and thermal applications [2]. To meet the demands from the electric power industry, the bolted Al–Cu joints have been substituted by welds.
Dissimilar metal joining is more difficult than joining two similar materials as they have different chemical, mechanical, and thermal properties. It is difficult to produce high quality Al–Cu dissimilar joint by fusion welding techniques as there exist a large variation of melting points, brittle intermetallic compounds and crack formation [4, 5]. Due to these reasons, the solid-state joining methods, such as friction welding, roll welding, and explosive welding are becoming more popular [6–8]. However, these methods also have various issues like friction welding and roll welding lack versatility, and explosive welding involves in the safety problems etc.

In the past two decades, FSW has developed to become an executable and significant welding process especially in aerospace and automotive applications involving Al alloys [9]. Welded joints can be used instead of riveted joints due to their lower production costs, better corrosion resistance and weight savings. Consequently, in recent time the FSW process has been known as a fundamental technology for fuselage and wing manufacturing by major aircraft manufactures [10]. FSW of dissimilar metals and alloys is becoming popular particularly for systems that are troublesome or impossible to weld by conventional fusion welding [11, 12]. FSW is a novel technique of joining materials, patented by “The Welding Institute” (TWI) UK, in 1991 [13]. In FSW, a non expandable rotating tool harder than the base metal (BM) is plunged into the abutting edges of the plates to be joined under sufficient axial force and advanced along the line of joint. During welding, the weld metal (WM) undergoes elevated temperature, severe plastic deformation and stress-strain course. However, the WM does not fuse, which supports to avoid several defects that are produced during fusion welding. The benefits of FSW process as a technology include: greater weld strength in compared to the fusion welding, little or no porosity, free from use of consumables, free from solidification cracks, free from affluent and no welding fumes or gases, no dependence on welder skill and lower cost of production. FSW has revolutionized the metal joining techniques due to its less energy consumption, environmental friendliness and versatility. FSW process is schematically shown in Fig. 1.

Joining of Al with Cu is difficult as their joining results in hard and brittle intermetallic compounds that reduce the joint strength, toughness and increase the electrical resistivity [15].

Xue et al. [5] studied the impact of welding parameters on surface morphology, interface microstructure and mechanical properties for butt joints of 1060 aluminum alloy and commercially pure Cu. There results revealed that when the stronger of the two materials i.e. Cu plate was placed at the advancing side sound joints were produced and good tensile properties were obtained at pin offsets of 2 and 2.5 mm. They also observed stacking layered structure at the Al–Cu interface under higher rotation rates. The impact of welding parameters on surface morphology, interface microstructure and mechanical properties on butt joints of AA 6063 Al alloy and commercially pure Cu was studied by Agarwal et al. [16]. Effectively good joints were produced when the stronger of the two materials (i.e. Cu plate)
was fixed at the advancing side. Fotouhi et al. [17] studied FSW butt joint of Al 5083 to commercially pure Cu and concluded that welding of the joint conducted at rotation speed of 800 RPM and tool traverse speed of 60 mm/min had the highest tensile strength (reportedly, about 98% of the weak base metal). They also detected intermetallic compounds in the stir zone (SZ). Friction stir welds (FSWs) between 5A02 aluminum alloy and pure copper were investigated by Tan et al. [18]. They reported the presence of exceptional metallurgical bonding between Al and Cu and attributed this to good tensile and bending strength. They also concluded that formation of layered microstructures caused an inhomogeneous hardness profile.

Confined research has been done on the FSW dissimilar Al-Cu joints till now, but still there continue to be a lack of systematic and methodical research. The Al-Cu dissimilar welding is expected to bear a substantial effect on the factors like pin offset, side of placement of base metal and plunge depth and a very less systematic studies have been reported in the literature. The AA 8011 H18 (good workability, good corrosion resistance and good electrical conductivity) and pure Cu 99.65% (high thermal and electrical conductivity) has been FSWed in this study. Microstructure analysis, tensile testing and microhardness test is performed on the joint thus produced and effect of plunge depth is investigated.

2. Experimental set up

Friction stir welds between 3 mm thick Al 8011 H-18 Al and pure Cu plates were produced in the department of mechanical engineering, Jamia Millia Islamia (a central university), New Delhi, INDIA on a robust vertical milling machine retrofitted to perform FSW. The plates were machined into the required size (200 mm x 50 mm). The chemical composition by weight (wt %) of Al 8081 and Cu plate is given in Table 1 and Table 2, respectively. The vertical milling machine used for performing the FSW experiments is shown in Fig. 2.

A non-consumable FSW tool with a tapered cylindrical pin and flat shoulder made of tungsten carbide was used to fabricate the joints as it comprises of out-
Table 1.
Composition of 8011 Al alloy according to spectrometer analysis (wt %)

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Cu</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Ni</th>
<th>Mn</th>
<th>Zn</th>
<th>Sn</th>
<th>Pb</th>
<th>Ti</th>
<th>Cr</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>98.50</td>
<td>0.103</td>
<td>0.086</td>
<td>0.231</td>
<td>0.0710</td>
<td>0.012</td>
<td>0.132</td>
<td>0.160</td>
<td>0.004</td>
<td>0.019</td>
<td>0.012</td>
<td>0.021</td>
<td>&lt;0.01</td>
</tr>
</tbody>
</table>

Table 2.
Composition of Cu according to spectrometer analysis (wt %)

<table>
<thead>
<tr>
<th>Element</th>
<th>Cu</th>
<th>Sn</th>
<th>Fe</th>
<th>P</th>
<th>Ni</th>
<th>Co</th>
<th>Ag</th>
<th>Zn</th>
<th>Other element</th>
<th>Other total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>99.65</td>
<td>0.0037</td>
<td>0.001</td>
<td>0.043</td>
<td>0.235</td>
<td>0.001</td>
<td>0.018</td>
<td>0.012</td>
<td>&lt; 0.001</td>
<td>&lt; 0.012</td>
</tr>
</tbody>
</table>

Fig. 2. FSW set up used for FSW experiments

standing high temperature strength, and high temperature wear resistance and good thermal conductivity. The tool used in the study comprised of shoulder diameter of 20 mm, pin of 6mm diameter at the root and 2.75 mm pin length. The rotating and simultaneously traversing tool with a conical pin having a tool tilt angle actually cause material movement akin to extrusion and forging action ahead and behind the tool, respectively. The tool with configuration effectively mixes the material during welding [14]. The welds were performed by keeping Cu on the advancing side and the Al-alloy on the retreating side. The weld runs were performed by keeping the tool 1mm towards the Al-alloy side. (i.e. 1 mm tool offset towards retreating side). The chemical composition of tungsten carbide (WC) tool is given in Table 3.

Table 3.
Composition of Cu according to spectrometer analysis (wt %)

<table>
<thead>
<tr>
<th>Element</th>
<th>WC</th>
<th>Ni</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>91.6</td>
<td>8.22</td>
<td>0.12</td>
</tr>
</tbody>
</table>
In view of a limited research to guide on dissimilar Al-Cu FSW welds, extensive trial runs were performed to identify the significant process parameters and obtain the working range of identified parameters. Based on the findings of trial runs it was decided to vary the spindle rpm and plunge depth during FSW experiments. The spindle rpm at levels of 560, 710, 900 and 1120 and plunge depth in the range of 0.1-0.4 mm varied in steps of 0.05 mm were varied. However, the welds produced at 710 rpm and plunge ranging between 0.1-0.25 mm were free from defects and showed good surface morphology. The final parameters that were selected for the study are given in Table 4.

<table>
<thead>
<tr>
<th>Process parameter</th>
<th>Final selected values</th>
<th>Fixed/Variable</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tool rotational speed</td>
<td>710 rpm</td>
<td>Constant</td>
</tr>
<tr>
<td>Tool tilt angle</td>
<td>2 degrees</td>
<td>Constant</td>
</tr>
<tr>
<td>Tool traverse speed:</td>
<td>100 mm/min</td>
<td>Constant</td>
</tr>
<tr>
<td>Plunge depth</td>
<td>0.1-0.25 mm</td>
<td>Variable</td>
</tr>
</tbody>
</table>

Metallographic and tensile samples were machined using a CNC Wire-EDM. The metallographic samples were polished and etched by a mixture of FeCl$_2$ (6 g) + HCl (10 ml) + H$_2$O (90 ml), in the Cu side whereas the Al side was etched using mixture of HF (1 ml) + HCl (1.5 ml) + HNO$_3$ (2.5 ml) + H$_2$O. The transverse sections of the samples were examined at through optical microscopy. The optical microscopy was performed at the mid depth of the transverse sections of the weld. Tensile tests of the specimens were prepared according to ASTM E8M and the tests were performed at room temperature at a crosshead speed of 2 mm/min. The micro-hardness profile across the weld bead was also measured at 0.2 N load using a microhardness testing machine (model – HM 200, Make – Mitutoyo, Japan).

3. Results and Discussion

3.1. Effect of stronger B M on advancing/retreating side

Literature reveals that when a strong and a soft material, like Cu and Al are FSWed, the resultant weld quality is significantly influenced by the fixed location [19, 20]. Fig. 3a and 3b shows the surface morphologies FSW Al–Cu joints for the different fixed locations at a welding parameter of 710 rpm – 100 mm/min. When the Cu plate was fixed on the advancing side, good sound weld surface was achieved, as shown in Fig. 3a. However, when the fixed location reversed (Cu plate was fixed on the retreating side) there was a severe lack of consolidation of joint and the weld was very poor (as seen in Fig. 3b). A clear separation (crack) of the two BM plates was visible through the weld surface. The occurrence as in Fig. 3 (a and b) can be described as that during the FSW process; the materials were
deposited from the retreating side to the advancing side behind the pin during the welding. When the Cu plate (stronger material) was placed at the retreating side, it was difficult to transfer to the advancing side because Cu hardly flew being a harder material. However, when the softer material (Al) was fixed at the retreating side, the soft material was easily transported to the advancing side, and the material flow cycle in the nugget zone was performed ordinarily.

![Fig. 3. Surface morphologies of the FSW Al-Cu joints under a welding parameter of 710 rpm – 100 mm/min the Cu plate fixed at (a) advancing side (b) retreating side](image)

### 3.2. Tensile Testing

#### 3.2.1. Tensile results

The tensile data of the welds produced at 710 rpm – 100 mm/min using 20 mm shoulder diameter tools are presented in Table 5. The tensile specimens were taken from each sample at all plunge depths ranging between 0.1–0.25 mm. Table 6 represents the tensile strength of the BM.

<table>
<thead>
<tr>
<th>Plunge depth (mm)</th>
<th>Tensile strength (MPa)</th>
<th>Fracture location</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>122.4</td>
<td>TMAZ of Al 8011</td>
</tr>
<tr>
<td>0.15</td>
<td>132.4</td>
<td>Nugget</td>
</tr>
<tr>
<td>0.2</td>
<td>137.6</td>
<td>TMAZ of Al 8011</td>
</tr>
<tr>
<td>0.25</td>
<td>114.3</td>
<td>TMAZ of Al 8011</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Base metal</th>
<th>Tensile strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA 8011 H18</td>
<td>127.3</td>
</tr>
<tr>
<td>Cu</td>
<td>350.4</td>
</tr>
</tbody>
</table>
3.2.2. Effect of plunge depth on tensile strength

The welds evidently rupture at the weakest part of the joint. The sub-size tensile test samples were machined with entire gauge lengths accommodated in the weld bead so as to assess the actual joint strength. The fractured tensile test specimens are shown in Fig. 4. Although the bead geometry features were not measured, the joint in the present case visibly appeared to have failed in the following locations (Fig. 4(a-d)) (i) near thermo-mechanically affected zone (TMAZ) of Al, (ii) Nugget Zone (NZ), (iii) near TMAZ of Al side and (iv) near TMAZ of Cu side. At excessively high plunge depth of 0.25 mm, the strength is less than the strength of Al and the fracture takes place near TMAZ of Cu side. This is notable, because of the thinning effect reduces the transport of materials on the advancing side behind the pin (which is also the fracture location). At plunge depth of 0.2 mm the fracture takes place near TMAZ of Al side. Typically, the fracture locations of the joints bear reliance on the microhardness distributions in the joints. Due to contrasting properties between the alloys, the microhardness distributions in the joints are different for the different alloys, thus resulting in the different fracture locations. A microhardness profile was consequently also studied to relate the strength to its hardness.

![Fracture location of the four samples](image)

The tensile test specimen from all the four experiment conditions specified in Table 5 was tested and the strengths of the four samples are depicted in the chart (Fig. 5). It is evident from the chart that the joint strength is equal to or greater than the strength of the weaker material accept for the plunge depth of 0.25 mm, further that the maximum strength is obtained at plunge depth of 0.2 mm. Low plunge depth, i.e. 0.10 mm, resulted in defected weld surface as insufficient material could flow through.
The increase in the plunge depth increases the axial thrust force, which in turn also increases the frictional heat, consequently leading to better material flow, proper mixing and joint consolidations [21]. With the joint formed at optimum heat input condition, the material is simultaneously severely plastically deformed and dynamically recrystallized in the NZ. This may be the reason for tensile strength of the joints to raise at higher plunge depth. On increasing the plunge depth beyond the optimum level (i.e., 0.2 mm), not only the thinning takes place due to reduced space under the shoulder, but also increases the associated heat input and the NZ temperature rises further, which in turn causes grain growth in an already dynamically recrystallized fine grained NZ [22]. As such, tensile strength decreases due to local thinning and grain coarsening.

### 3.3. Microhardness profiling results

#### 3.3.1. Microhardness variation across the weld

Microhardness profile, shown in Fig. 6, evidently shows decrease in microhardness values at the retreating side TMAZ compared to other regions across the weld, thus resulting in fracture.

The average Vickers microhardness (HV\textsubscript{0.2}) values of the parent materials – Al Alloy and Cu are 55 and 100, respectively. It was observed that in all the welds the higher HV values in a range of between 118 and 190 were measured in the TMAZ and NZ on the Cu side. NZ of Al side was having microhardness values maximum up to 130. The microhardness variation was, moreover, similar in the Heat Affected Zone (HAZ) and TMAZ in Al region ranging from 55–65. The microhardness variation was low in the HAZ regions of Cu. The microhardness profile also reveals sudden large peaks at the interface of the welds that are characteristic of the presence of intermetallics compounds typically formed during Al-Cu dissimilar welds [15].
Fig. 6. Microhardness profiles of welds produced at a constant spindle speed of 710 rpm with the shoulder diameter 20 mm at varying plunge depth.

Fig. 7 shows the variation of hardness from top to bottom at centre of weld nugget. It was observed that the hardness of weld nugget at the top was higher than at the bottom. This instability of hardness from top to bottom of the weld nugget can be imputed to variation in grain size, strain hardening effects, the fraction of copper at the top region is higher than that of other areas and due to intermetallic compounds. Similar kind of results was also reported by Won-Bae Lee [23] during welding of copper plates. The material closest to the tool shoulder undergoes greater plastic deformation as compared to material that is away from the tool, thus leading to refine grain structure.

3.3.2. Effect of plunge depth on microhardness

Figs. 6 and 7 show the variation of microhardness with plunge depth. Highest values of the microhardness are visible for 0.2 mm plunge depth as compared to other plunge depth values. This variation bears the similarity with the variation of the tensile strength. Like the microhardness, the tensile strength of the joint...
with plunge depth 0.2 mm is also high. At low plunge depth of 0.1 mm, causing the heat to remain insufficient the microhardness and the strengths both are low. This corroborates the fact that an increasing plunge depth causes increase in the heat input becoming sufficient for the adequate softening of the material, proper mixing and joint consolidation. Under these conditions, the grains also dynamically recrystallized and refined due to severe plastic deformations. Thus, finer grains are formed at the weld nugget fabricated under 0.2 mm plunge depth leading to increase microhardness and strength. As plunge was further increased after 0.2 mm due to unstable material flow, local thinning in the weld nugget took place.

3.4. Microstructural evaluation

3.4.1. Microstructure in the weld nugget

The microstructure zones characteristic to the FSW were identified in all the welds using an optical microscope. The weld made with 0.2 mm plunge depth was analyzed for microstructural appearance as they have highest tensile strength and microhardness.

![Micrographs of weld by optical microscopy at 500X, (a) Al NZ (b) Cu NZ](image)

A characteristic mixed and asymmetric structure on the NZ of Al side was found (Fig. 8a) and lamellar alternating structure characteristic was seen in the NZ at the Cu side (Fig. 8b). During FSW, the peak temperature (i.e. the temperature of NZ) is lower than the solidus temperature of either Al or Cu. Also, peak temperature on the Al-alloy side is very high as compared to Cu the side (due to vast difference the melting point of the two). The grains in the NZ region on Al side were evidently refined and would have experienced a “continuous” dynamic recrystallization (CDRX) [24]. However, as the temperature is expected to remain at the lower limit of the recrystallization temperature for Cu, it doesn’t undergo the CDRX process. Consequently, the NZ on the Cu side appeared to comprise
of lamellar alternating structure. Additionally, there also exists difference in heat conductivities of Al and Cu. Owing to higher heat conductivity of Cu than Al, frictional heat produced by stir action in Cu side is expected to be conducted more into the nearby metal than in Al side. Due to this fact, temperature gets reduced in the NZ leading to different structure on different sides of NZ. Hence, no obvious metallurgical process would be produced between Al and Cu during the FSW. Therefore, the Cu and Al were closely bonded in the Al side showing a mixed structure.

Fig. 9 shows the enlarged view of the NZ made up of characteristic alternate lamellae of dark and light construction that may comprise of Cu/Cu$_9$Al$_4$. The bright region represents unmixed Cu lamellae with a hardness range of 95-100 HV$_{0.2}$, while the dark region appear to be a typical Cu-rich phase mixed with some quantity of Al by pin stirring action. The dark regions has a hardness range of 120-185HV$_{0.2}$, which may contain certain percentage of the Cu$_9$Al$_4$ intermetallic compound, as it is obvious that the interface of solid state welded Al/Cu is prone to growth of intermetallic compounds at temperatures above 120°C [15]. This presumption gains supports from similar results reported by OUYANG et al. [25]. They reported the formation of Cu$_9$Al$_4$ intermetallic compound in the nugget having hardness value in the range of 136-178 HV$_{0.2}$. The evolution for Cu$_9$Al$_4$ structure is reported to be due to 1) mechanical mixing due to stirring action of the tool pin 2) precipitate dissolution 3) interdiffusion in the grain boundaries. Similar results of phase Cu$_9$Al$_4$ were also reported by Aritoshi [26].

![Fig. 9. Enlarged views of lamellar structure in weld nugget close to Cu side at 1000X magnification](image)

3.4.2. Effect of plunge depth on microstructure

Micrographs were taken at different regions but for the comparison purpose, the micrographs of SZ and TMAZ regions in Al side and Cu side are shown in Fig. 10 and Fig. 11. From the micrographs, it is seen that there is a notable variation in grain sizes of different zone microstructure.
The grains in the NZ are finer than those in TMAZ. The grains are relatively finer at NZ and TMAZ of the joint fabricated under plunge depth of 0.2 mm (Figs 10, 11 (c, d)). However, the grains are relatively larger at the SZ and TMAZ of the joints fabricated under plunge depth of 15 mm (Fig. 10, 11 (a-d)) and 0.25 mm (Fig. 10, 11 (a-d)). Under low plunge depth, the fragmentation of grains is less due to low axial force, which subsequently results in coarse grains in weld nugget region. With increase in plunge depth, the frictional force increases leading to increase in heat input to the weld. The increased heat input raises the nugget zone temperature and this could lead to static grain growth of dynamically recrystallized
grains, which may be the reason for finer grains under plunge depth of 0.2 mm. When plunging depth increases to 0.25 mm, the heat generation is more but the cooling rate of the weld nugget diminishes gradually leading to grain coarsening.

4. Conclusion

1. Successful FS welds between Al/Cu with limited intermetallic compounds were produced. Tool rotation speed of 710 rpm; plunge depth of 0.2 mm and transverse speed of 100 mm/min, were found to be the most appropriate to join Al and Cu.
2. Tensile results show that highest tensile strength obtained is at a plunge depth of 0.2 mm, which is higher than the strength of the Al base metal. The strength of the joint decreased with increase in the plunge depth after 0.2 mm.

3. The joint failed in any one of the given location (i) weld zone, (ii) near TMAZ of AA 8011 side and (iii) near TMAZ of Cu side. This was related to decreased microhardness in TMAZ, defects in advancing side and intermetallics in weld nugget.

4. There is a comparable variation in grains of different zone microstructure. The grains are relatively finer at NZ and TMAZ of the joint fabricated under plunge depth of 0.2 mm.

5. A characteristic mixed and asymmetric structure on the NZ of Al side was found, as shown in figure and lamellar structure characteristic was seen in the nugget at the Cu side.

Acknowledgements

The authors thank the University Grant Commission (UGC) for its financial assistance (vide sanction order no. F.3-40/2012(SAP-II) under its sap (DRS-I) sanctioned to the Department of Mechanical Engineering for the project entitled Friction Stir Welding and Ultrasonic Machining.

Manuscript received by Editorial Board, January 11, 2016; final version, July 11, 2016.

References


FRICTION STIR WELDS OF Al ALLOY-Cu


Zgrzewanie tarczowe z przemieszaniem stopu aluminium z miedzią. Badanie wpływu zagłębień trzpienia

S t r e s z c z e n i e

W pracy przedstawiono spawane czołowe aluminium (Al) 8011-H18 i czystej miedzi (Cu) wykonane techniką spawania tarczowego z przemieszaniem (FSW). Badano wpływ zagłębień trzpienia na morfologię powierzchni, mikrostrukturę i właściwości mechaniczne spawu. Spawy wykonano przy zmiennym zagłębienniu trzpienia narzędzia w zakresie od 0,1 do 0,25 mm. Złącza bez defektów otrzymano, gdy płyta miedziana była zamocowana po stronie natarcia przesuwu. Stwierdzono, że przy mniejszym zagłębienniu narzędzia uzyskuje się większą wytrzymałość na rozciąganie niż przy większych zagłębieniach, przy których występuje lokalne pocienienie w regionie spawania. Dobre właściwości uzyskano przy zagłębiieniu 0,2 mm, a wytrzymałość spawu na rozciąganie była większa niż wytrzymałość aluminium (słabszego z dwu metali). Badanie mikrostruktury ujawniło, że metal po stronie miedzi miał w strefie rekryystalizacji (strefie ziarnej) spawu (WNZ) strukturę płytkową o charakterze przemiennym. Natomiast mieszana struktura Cu i Al istniała w strefie rekryystalizacji po stronie aluminium. Wyższe wartości mikrotwardości były obserwowane w obszarach międzyfazowych złącza, co wynikało z odkształceń plastycznych i obecności związków międzymetalicznych.