DIRECT DIFFUSION WELDING OF Fe_3Al TO STEEL

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الملخص

تم وصل ألومينيد الحديد (Fe_3Al) والفولاذ بنجاح باستخدام طريقة اللحام بالانتشار المباشر في فرن مفرغ من الهواء وأجريت تجارب اللحام بالانتشار عند درجة حرارة وصل من 1000م^o إلى 1200م^o ، وعند أوقات وصل من 10 إلى 60 دقيقة وتحت ضغط ثابت مقداره 10 ميغابسكال. وتم تشكيل وصلات الفولاذ /Fe_3Al وتم تحليل معالم البنية المجهرية لوصلات الفولاذ /Fe_3Al بتقنيات وصف متنوعة مثل المجهر الضوئي والمسح باستخدام المجهر الالكتروني المزود بمطياف لقياس الطاقة المتشتتة ومسبار التحليل المجهري الالكتروني وكذلك باستخدام مقياس حيود الأشعة السينية وتم قياس الصلادة المجهرية من Fe_3Al إلى الفولاذ باستخدام جهاز فيكر لقياس الصلادة المجهريه. كما تم قياس قوة القص لوصلات الفولاذ/Fe3Al باستخدام آلة اختبار القص، حيث بلغت أقصى قوة قص لوصلات الفولاذ/Fe_3Al 362 ميغابسكال لوصلات وصلت عند درجة حرارة وصل 1100م^o، وعند وقت وصل 60 دقيقة. وظهرت مناطق التحول بوضوح في المنتصف بين Fe_3Al والفولاذ وبلغ أقصى عرض لمنطقة التحول حوالي 159 ميكرومتر لوصلات الفولاذ/Fe_3Al والتي وصلت عند درجة حرارة وصل 1100م^o وعند وقت وصل 30 دقيقة.

مقياس الحيود يظهر وجود طور جديد على سطوح وصلات الفولاذ/Fe3Al والملحومة باستخدام طريقة الوصل بالانتشار وهو Fe_3AlC بالإضافة إلى الأطوارٍ الأخرى وهي Fe_3Al, FeAl, α-Fe وعدم وجود أطوار هشة لوصلات الفولاذ/Fe_3Al والتي وصلت عند درجة حرارة وصل 1100م^o، وعند وقت وصل 60 دقيقة وبلغت قيمة الصلادة المجهرية عند منطقة التحول بالقرب من جهة الفولاذ حوالي (189 HV) وازدادت إلى حوالي (301 HV) بالقرب من جهة Fe_3Al.

ABSTRACT

Iron aluminides Fe_3Al intermetallics and steel were joined successfully by vacuum diffusion bonding. Diffusion bonding experiments were carried-out at different bonding temperature of 1000, 1100 and 1200 ^{0}C and at different bonding time of 10, 30 and 60 minutes. The bonding pressure was 10 MPa. Fe_3Al/steel joints were thus formed. The microstructure features in the Fe_3Al/steel joints were analyzed by a variety of characterization techniques such as optical microscopy, scanning electron microscopy (SEM) equipped with energy-dispersive spectroscopy (EDS), electronic probe microanalysis (EPMA) and an X-ray diffraction technique. The micro-hardness from Fe_3Al intermetallics to steel were measured with Vickers microhardness tester. The shear strength of the Fe_3Al/steel joints was evaluated with a shear test machine. The maximum shear strength of Fe_3Al/steel joints was 362 MPa for joints bonded at 1100 ^{0}C and bonding time of 60 min. Transition zones in the middle between the Fe_3Al intermetallic and the steel were observed and the width of the transition zone was 159 µm for Fe_3Al/steel joint bonded at bonding temperature of 1100 ^{0}C and bonding time of 30 min. X-ray diffraction results showed the existence of new phases such as aluminum iron carbide (Fe_3AlC) and FeAl with steel (α-Fe) and Fe_3Al, in the Fe_3Al/steel diffusion-welded joint without brittle phases and the micro-hardness values at the

transition zone increased from about 189 HV close to steel side to about 301 HV close to Fe_3Al side.

KEYWORDS: Fe_3Al/steel diffusion welded joint; microstructure; micro-hardness; shear strength; EPMA and x-ray diffraction.

INTRODUCTION

Aluminides possess sufficiently high concentrations of aluminum to form a continuous fully adherent alumina layer on the surface when exposed to air or oxygen atmospheres. The amount of aluminum present in aluminides can range from 10 to 30 wt% and is significantly higher than the aluminum concentrations present in conventional alloys and superalloys. In the case of nickel and iron aluminides, the alumina layer formed on the surface of the materials is responsible for their excellent oxidation and carburization resistances even at temperatures as high as 1000°C or higher. Therefore, aluminides, unlike conventional steels and superalloys based on nickel, iron, and cobalt do not necessarily require chromium to form an oxide layer on the surface of the material to be protected against high-temperature oxidation and corrosion. Alumina is much more thermodynamically stable at high temperatures than Cr_2O_3. In addition, their oxidation and carburization resistances, aluminides possess lower densities, high-melting points, and exhibit interesting mechanical properties due to their ordered crystal structures. The strength of some intermetallics increases with temperature instead of exhibiting a decrease; thus, they are ideally suited for high-temperature applications [1]. These advantages lead to several potential uses including heating elements, furnace fixtures, heat-exchanger piping, sintered porous gas-metal filters, automobile and other industrial valve components, catalytic converter substrates and components for molten salt applications [2].

Rotary friction welding has been used to successfully join Fe_3Al to steel. The tensile strength was 75 MPa for Fe_3Al/steel joint bonded at friction time of 2700 ms; pressure of 0.25 MPa and pressure in swelling phase of 0.35MPa [3]. Joining of Fe_3Al to Q235 carbon steel, firmly combined diffusion transition zone can be formed near the interface of Fe_3Al/Q235 dissimilar material by vacuum diffusion bonding under the condition of heating temperature T_b= 1050-1080°C, holding time t = 60 minutes and pressure of 9.8 MPa. There was an obvious diffusion characteristic in the transition zone. Fe_3Al and Q235 carbon steel were joined successfully at 1080 ^{0}C for 60 min by vacuum diffusion welding technology [4]. The experimental results indicated that Al atom content decreases from 27 to 1% and Fe atom content increases from 73 to 96% from Fe_3Al base metal to Q235 carbon steel [5]. The micro-hardness was 420–200 HM (microsclerometer hardness) and the shear strength was 71.4 MPa in the Fe_3Al/Q235 joint. There were Fe_3Al, α-Fe(Al) solid solution and FeAl phases with high micro-hardness in the Fe_3Al/Q235 diffusion-welded joint near the diffusion transition zone without brittle phases (such as $FeAl_2$, Fe_2Al_5, $FeAl_3$ etc.). This is favorable to improve the ability to resist cracking of the Fe_3Al/Q235 diffusion bonding joint. Thus, the ideal Fe_3Al/Q235 joint can be obtained, which will meet the requirement for engineering structures [4,5].

EXPERIMENTAL

Fe_3Al was prepared by vacuum induction melting and casting in vacuum furnace. The chemical composition of the Fe_3Al intermetallic was identified (Wt. %) to be 86.028% Fe, 13.87% Al, 0.1% Zr and 0.002% B. The chemical compositions of carbon steel (Wt. %) was: 0.16% C, 0.01% Si, 0.49% Mn and 99.23% Fe. Cutting of the Fe_3Al intermetallic and the steel rods in the form of discs of 20 mm diameter and 4mm thick for Fe_3Al intermetallic samples and of 16 mm diameter and 4 mm thick for steel samples. The samples were ground on different emery paper of grades, 100, 320, 600, 1000 and until 1200. The samples were rough polished by using alumina. After cleaning, the Fe_3Al sample was sandwiched between two steel samples, and then the assembly was positioned in the inductor coil of bonding apparatus. The experiments were carried out at a compressive strength of 10 MPa. The samples were heated to a temperature of 400^0C for 20 minutes to clean outside surfaces from contaminations. The samples were then heated to the required joining temperature (1000 to 1200^oC) and held at that temperature until the end of joining time (10 to 60 min). At the end of joining time, the samples were cooled to room temperature in the vacuum chamber at cooling rate of 10^0C/min. The vacuum in the vacuum chamber during the joining experiments was held between 1-5x10^{-3} Pa. Thus, the Fe_3Al/steel diffusion-welded joint was formed. After joining processes have been completed, the specimens were cut perpendicular to the joint transition zone. The specimens were mounted in plastic mould for easy handle of the specimens. The specimens were grinded on a series of emery papers (100, 220, 320, 600, and finally 1200) containing successively finer abrasives (silicon carbide). Polishing of specimens were done with polishing machine which was covered with a special leather that charged with carefully sized (less than 1 µm) abrasive particles (alumina) in order to remove the fine scratches. Finally, Steel etched by: 5 % nital and intermetallic etched by: 40 ml HCl + 30 ml HNO_3 + 20 ml glacial acetic acid + 10 ml glycerol. The microstructure in the Fe_3Al/steel diffusion-welded joint was observed by means of an optical microscope Neophot-21 type (OM) and scanning electron microscopy (SEM) equipped with electron probe X-ray microanalysis (EPMA) model JEOL JXA-50A type. The analysis parameters were:

Accelerating voltage = 2 Kv.	Range of penetration = 1.0-4.0 µm.
Absorption current I_A = 3x10^{-8} A.	Take-off angle = 35^0.

Energy dispersive X-ray analyzer (EDX) Oxford Instruments type ISIS with SiLi detector was used with DSM to investigate the distribution of elements across the transition zone of Fe_3Al/steel joints.

The microhardness from Fe_3Al to steel was measured by Vickers microhardness tester, with a test loading of 100 g and a loading time of 10sec. Three series of microhardness values were measured along three lines started from Fe_3Al to steel along the transition zone on the polished surface of Fe_3Al/steel sample. After that the average microhardness values were calculated at the transition zone in both sides and along the transition zone, Fe_3Al and steel.

The shear strength was evaluated with a shear testing machine (Instron TT 11-15 TYPE). The phases formed in the Fe_3Al/steel joint were analyzed with an x-ray diffractometer used is a Philips type. The main operating parameters are:

Scanning step = 0.05^o	Integrating time= 3 sec
Range (2θ) = 20-120^o	Voltage = 35 Kv
Current = 25 mA	

The target source of the X-ray was made from copper and the filament made from tungsten, $K_{\alpha1}$= 1.540562, $K_{\alpha2}$= 1.544390, $K_{\alpha3}$= 1.541638, and the penetration depth of the X-ray between 5-30 µm.

RESULTS AND DISCUSSIONS

Shear strength

Fe_3Al intermetallic was bonded to steel by direct diffusion bonding. The highest shear strength value obtained when Fe_3Al bonded to steel was 362 MPa for joints bonded at bonding temperature of 1100 ^{0}C and bonding time of 60 minutes. Bonding parameters and shear strength results of Fe_3Al/steel joints are listed in Table (1).

Table 1: Bonding parameters and average shear strength results of Fe_3Al/steel joints.

Sample No.	T_b^* (^{0}C)	t_b^* (min)	P^* (MPa)	Result	Ave. shear strength (MPa)
1	1000	30	10	B^*	304
2	1100	30	10	B	321
3	1200	30	10	B	278
4	1100	10	10	B	305
5	1100	60	10	B	362

*T_b= bonding temperature; t_b= bonding time; P= bonding pressure and B= bonded.

Figure (1) shows the effect of joining temperature on the joining strength of the Fe_3Al/steel joints. It can be seen from the figure that when the joining temperature was lower than 1100^{o}C, the joint strength increased with increasing the bonding temperature and reached its highest value 321 MPa for joints bonded at bonding temperature 1100^{o}C; for 30 min at bonding pressure of 10 MPa.

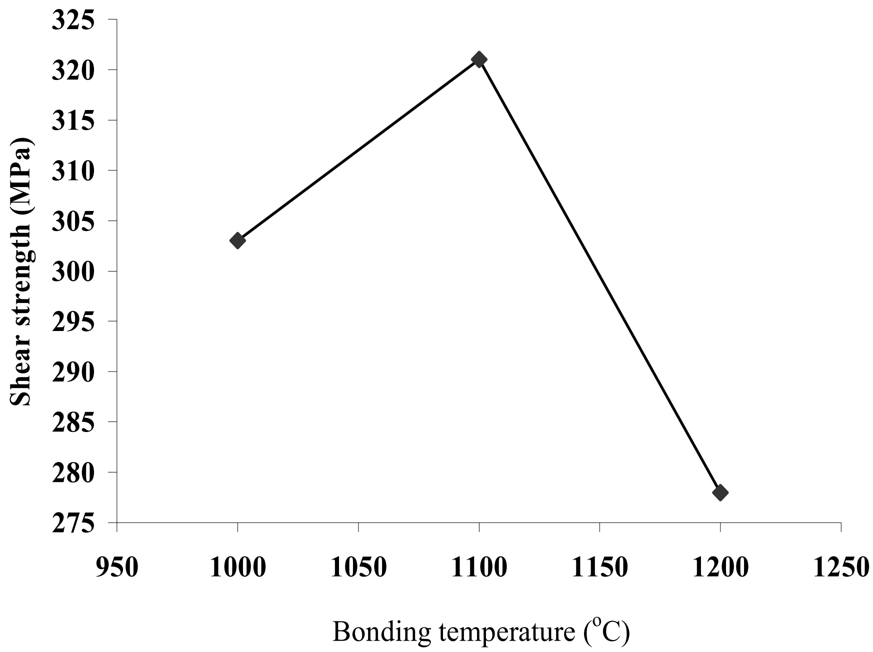


Figure 1: The temperature dependence of the shear strength of Fe_3Al/steel joints bonded at: t_b=30min; P=10 MPa.

However, the joint strength decreased when the joining temperature was higher than 1100^{o}C to 278 MPa for joints bonded at 1200^{o}C, for 30 min at bonding pressure of 10 MPa. Figure (2) shows the effects of holding time on the joint strength for Fe_3Al/steel joints bonded at 1100 ^{0}C and bonding pressure 10 MPa. From the figure, we can be seen that as the bonding time increased the joint strength increased and reached 362 MPa with joints bonded for 60 min.

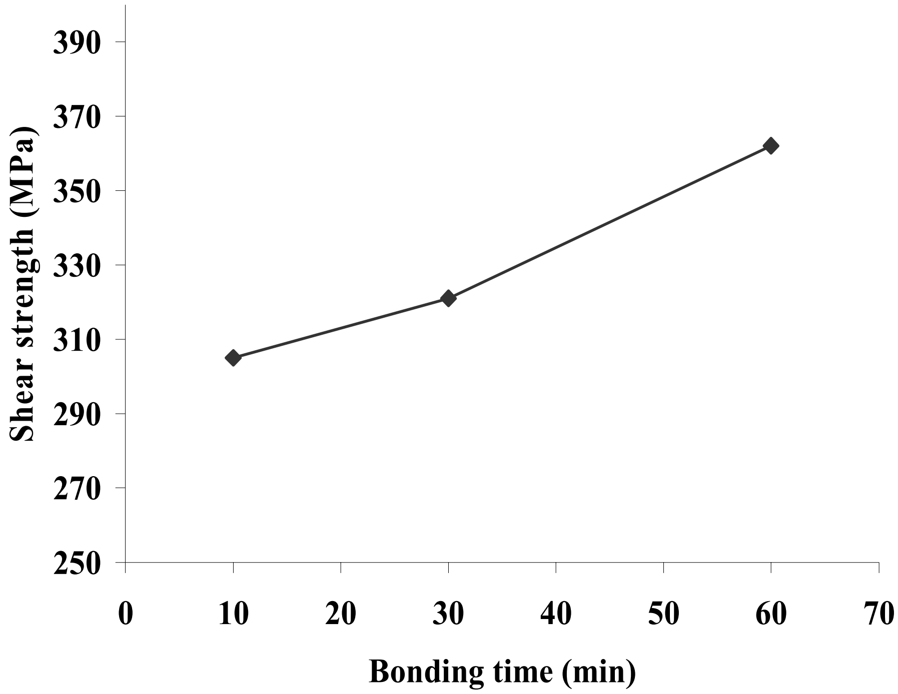


Figure 2: The effect of holding time on the joint strength of Fe_3Al/steel joints bonded at: T=1100 ^{0}C; P=10 MPa.

Micro-hardness results

The micro-hardness from steel to Fe_3Al across the Fe_3Al/steel joint was measured in order to study the performance in the whole Fe_3Al/steel diffusion welded joint. Figure (3) presents the microhardness measurements for Fe_3Al/steel joint bonded at 1100^{o}C; for 60 min at bonding pressure of 10 MPa. From the figure we can see that the average microhardness increased from about 189 HV at the transition zone close to steel side to about 301 HV close to Fe_3Al side while along Fe_3Al intermetallic compound, the average microhardness value of about 334 HV.

Figure (4) presents the microhardness measured values for Fe_3Al/steel joint bonded at 1200^{o}C; bonding time of 30min and bonding pressure of 10 MPa. From the figure it can be seen that the microhardness increased from about 118 HV at the transition zone close to steel side to about 233 HV at the transition zone closed to Fe_3Al intermetallic, while along the Fe_3Al intermetallic compound, the average micro-hardness value of about 255 HV. From the results of the microhardness, we can see that the average microhardness value at the transition zone of the Fe_3Al/steel joint was about 202 HV when the joining temperature was 1100^{o}C; bonding time 30 min and bonding pressure 10 MPa. When the joining temperature was 1200^{o}C; bonding time 30 min and bonding pressure 10 MPa, the average microhardness value of the transition zone closed to Fe_3Al intermetallic was about 206 HV.

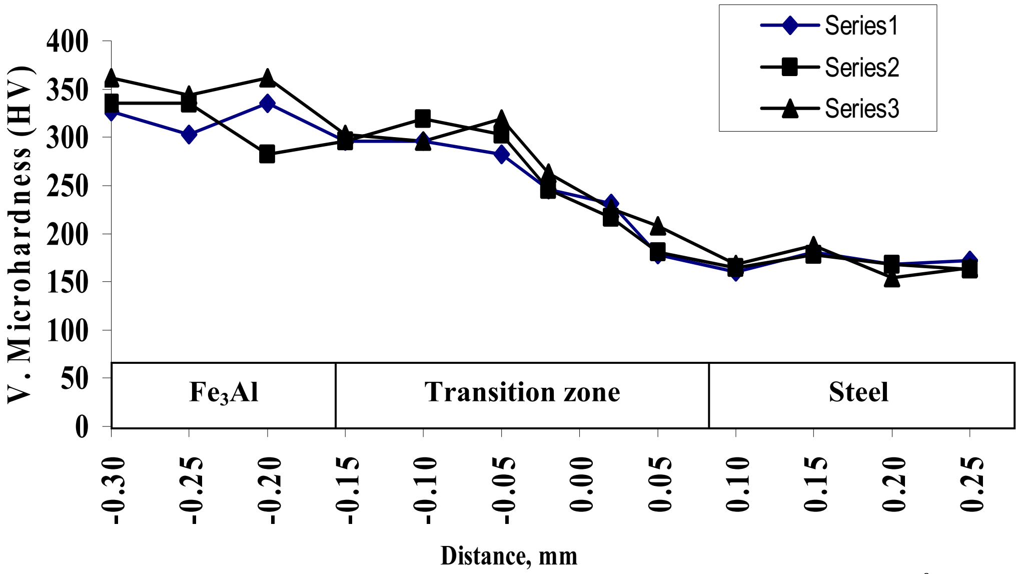


Figure 3: Vickers Microhardness of Fe_3Al/steel joint bonded at: T=1100 ^{0}C; t_b= 60 min. and P= 10 MPa.

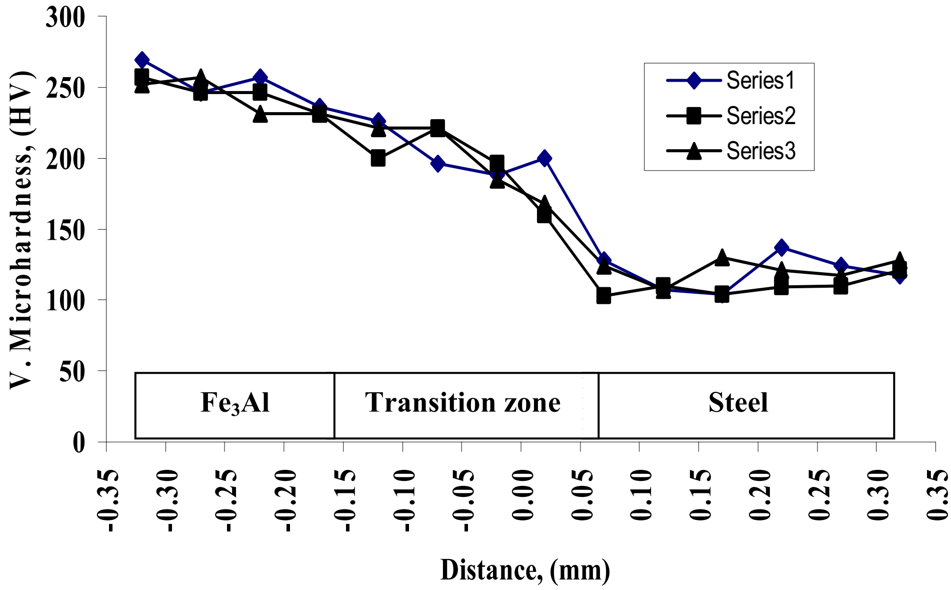


Figure 4: Vickers Microhardness of Fe_3Al/steel joint bonded at: T=1200 ^{0}C; t_b= 30 min. and P= 10 MPa.

Metallography study

The results of the metallographic samples of Fe_3Al/steel diffusion-welded joints were examined by using optical microscope. Figure (5) shows the microstructure of Fe_3Al and Figure (6) shows the microstructure of steel. The effects of bonding temperature (1100-1200^{o}C) on the microstructures of Fe_3Al/steel joints bonded for 30 min at bonding pressure 10 of MPa are shown in figures 7 and 8 respectively. The upper part is the Fe_3Al intermetallic compound and the lower part is the steel. In the middle is the transition zone. A diffusion transition zone was obviously observed. The width of the transition zone was 159 μm for joint bonded at 1100 ^{0}C as can be seen in Figure

(7b). However, the width of the transition zone decreased to 136 µm when the bonding temperature was increased to 1200^{o}C as can be seen in Figure (8b). Micro-voids were formed only during diffusion bonding of Fe_3Al to steel at 1200^{o}C; for 30 min and bonding pressure of 10 MPa as can be seen in Figure (8). During diffusion bonding the irregularities of the surface flatten into a grain boundary at the points of contact with micro-voids. Under the application of pressure and heating, Fe and Al atoms begin to diffuse into each other and the grain boundary and micro-voids disappear. But there retain a few of micro-voids at the transition zone near the side of steel, this explained why shear strength was decreased to 278 MPa for joints bonded at 1200^{o}C; while at 1100^{o}C; was 321 MPa.

The effects of bonding time on the microstructures of Fe_3Al/steel joints bonded at bonding temperature 1100^{o}C are shown in figures 9 and 10. In Figure (9), the upper parts are steel and the lower parts are Fe_3Al intermetallic compound while in Figure (10), the upper parts are Fe_3Al intermetallic compound and the lower parts are steel. A transition zone was obviously observed in the middle. It can be seen that the width of the joint transition zone first increased and then decreased with increment of holding time.

The width of the transition zone was 98 µm for joint bonded at bonding time of 10 min as seen in Figure (9b). However, the width of the transition zone increased when the bonding time was higher than 10 min and reached 159 µm for joints bonded at bonding time of 30 min as seen in Figure (7b). Further increase in bonding time result a decreased in the width of the transition zone and reaches 119 µm for joint bonded at bonding time of 60 min as seen in Figure (10b).

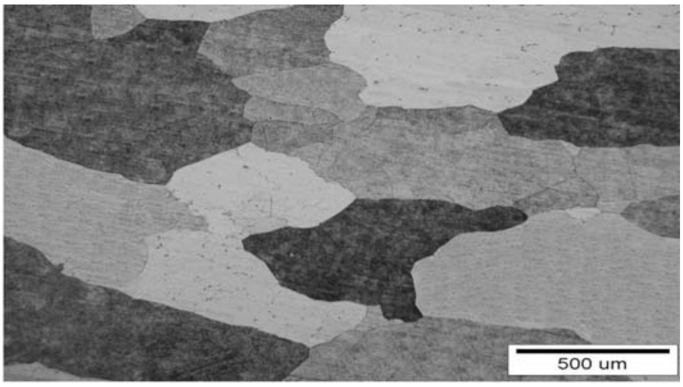


Figure 5: Optical micrograph of Fe_3Al.

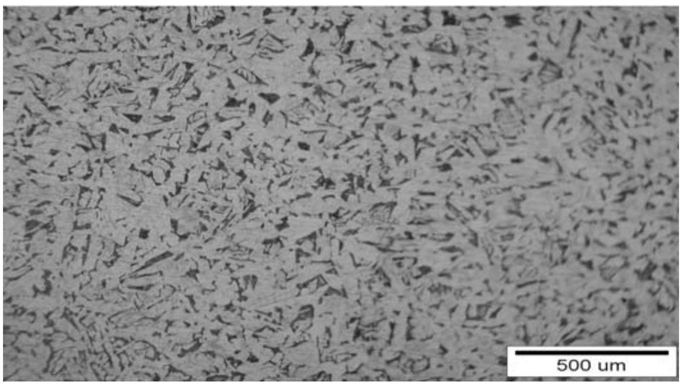


Figure 6: Optical micrograph of steel.

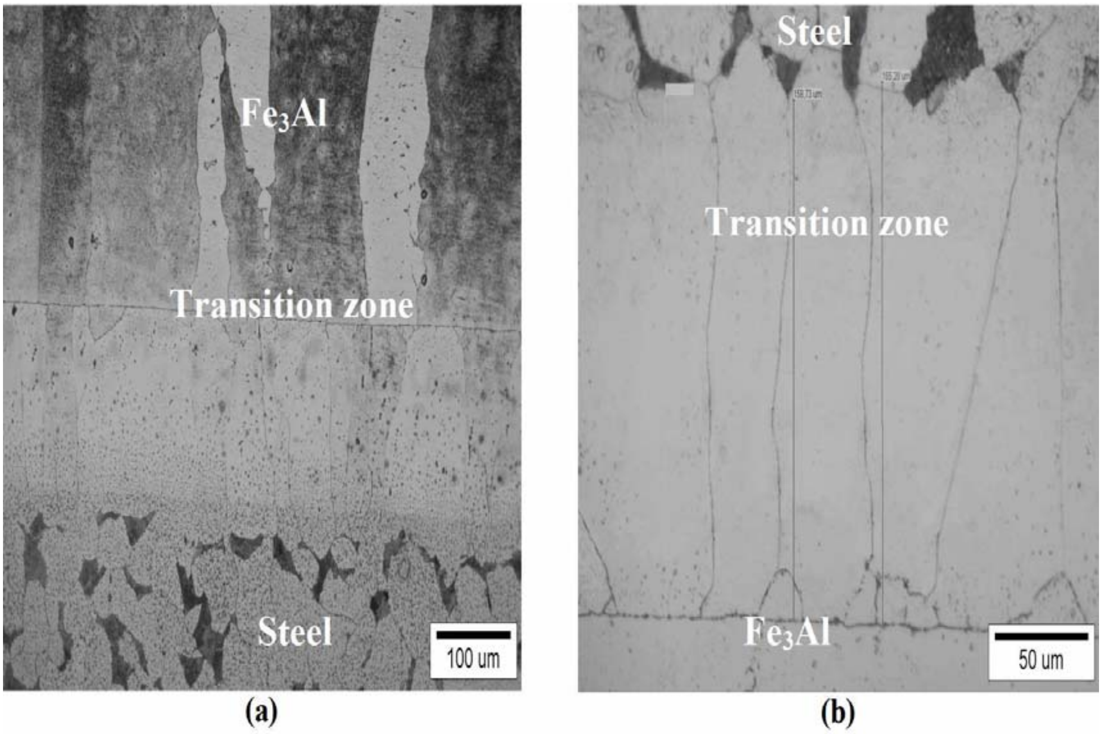


Figure 7: Optical micrograph of cross-section of Fe_3Al/steel joint bonded at: T=1100°C; t_b= 30 min; and P= 10 MPa: (a) x200; (b) The transition zone thickness measurements (159 µm) at x500.

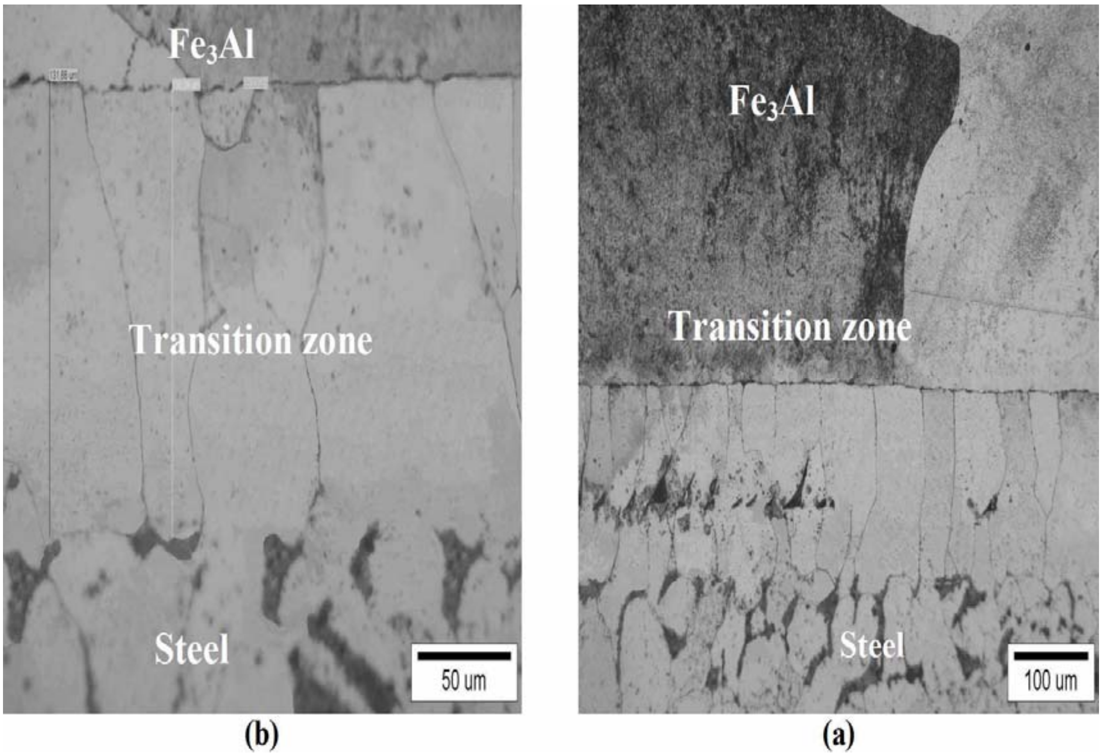


Figure 8: Optical micrograph of cross-section of Fe_3Al/steel joint bonded at: T=1200°C; t_b= 30 min; and P= 10 MPa: (a) x200; (b) The transition zone thickness measurements (136 µm) at x200.

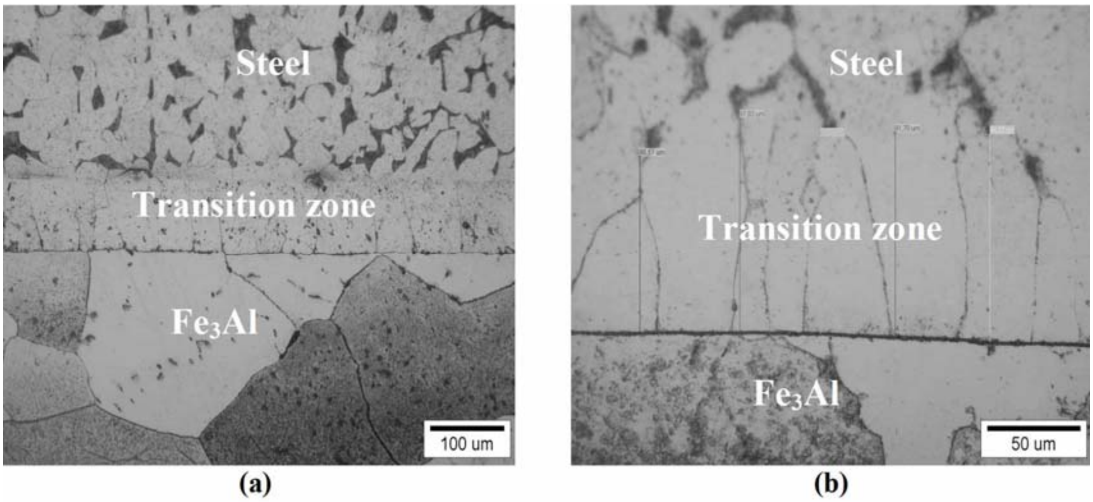


Figure 9: Optical micrograph of cross-section of Fe_3Al/steel joint bonded at: T=1100^oC; t_b= 10 min; and P= 10 MPa: (a) x200; (b) The transition zone thickness measurements (98 µm) at x500.

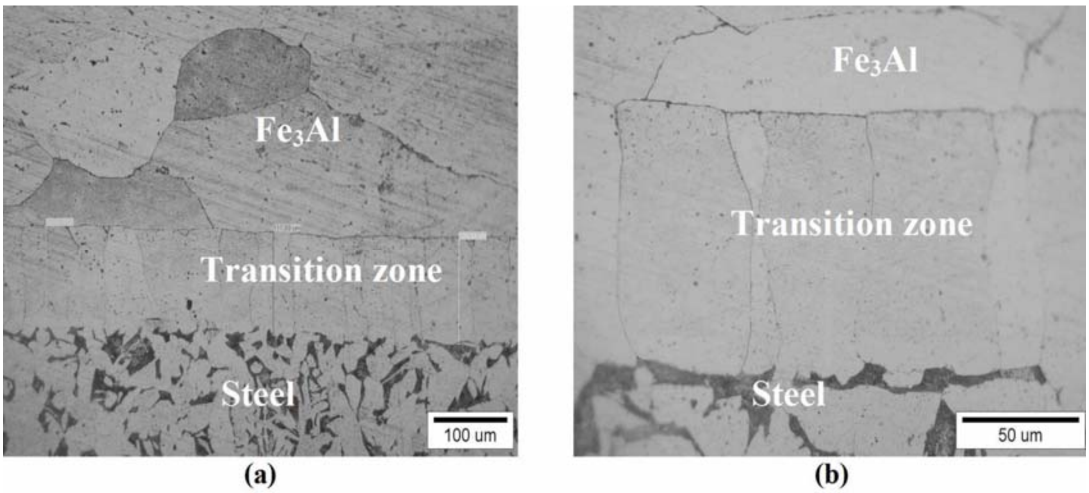
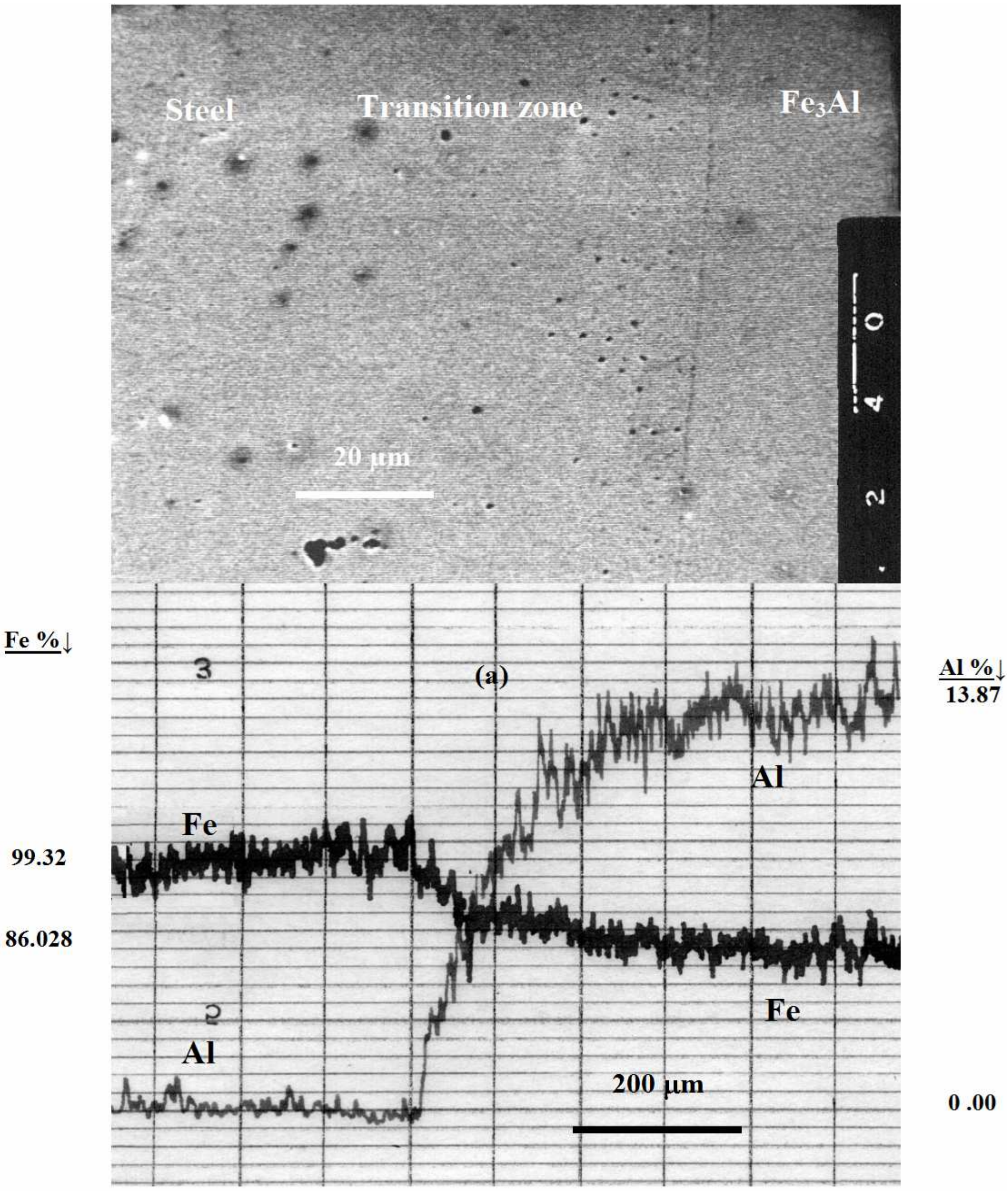


Figure 10: Optical micrograph of cross-section of Fe_3Al/steel joint bonded at: T=1100^oC; t_b= 60 min; and P= 10 MPa: (a) The transition zone thickness measurements (119 µm) at x200. (b) x500.

EPMA

The results of EPMA indicate linear distributions of Al and Fe across the Fe3Al/steel joints were illustrated in Figures (11 and 12) for joints bonded at different process parameters. Figure (11) shows linear distribution of elements of joint bonded at 1100^oC; for 60 min and bonding pressure of 10 MPa. It is clear that Fe concentration is regular along the steel, when reached the transition zone the concentration gradually decreased during crossing the transition zone and when passing from the transition zone to Fe_3Al intermetallic the concentration was regular. However, Al concentration was nil during passing along the steel, when reached the transition zone the concentration gradually increased during crossing the transition zone and when crossing the transition zone to Fe_3Al intermetallic, the concentration was regular.

Figure (12) shows linear distribution of elements Al, Fe; Mn and Zr of Fe_3Al/steel joint bonded at 1200 ^0C; for 30 min and bonding pressure 10 MPa. The figure shows that Fe concentration was regular along the steel and when reached the transition zone this concentration gradually decreased when crossing the transition zone and when passing from the transition zone to Fe_3Al intermetallic the concentration was regular. Al concentration was nil during passing along the steel, when reached the transition zone the concentration increased when crossing the transition zone and when passing from the transition zone to Fe_3Al intermetallic the Al concentration was regular.



(b)

Figure 11: EPMA line analysis across the Fe_3Al/steel joint bonded at: T=1100 ^{0}C; t_b= 60 min. and P= 10 MPa: (a) View of the transition zone with line of microanalysis. (b) Distribution of Al and Fe elements across the transition zone.

Mn and Zr concentrations are nil during passing along the steel and the transition zone and also along the Fe_3Al intermetallic.

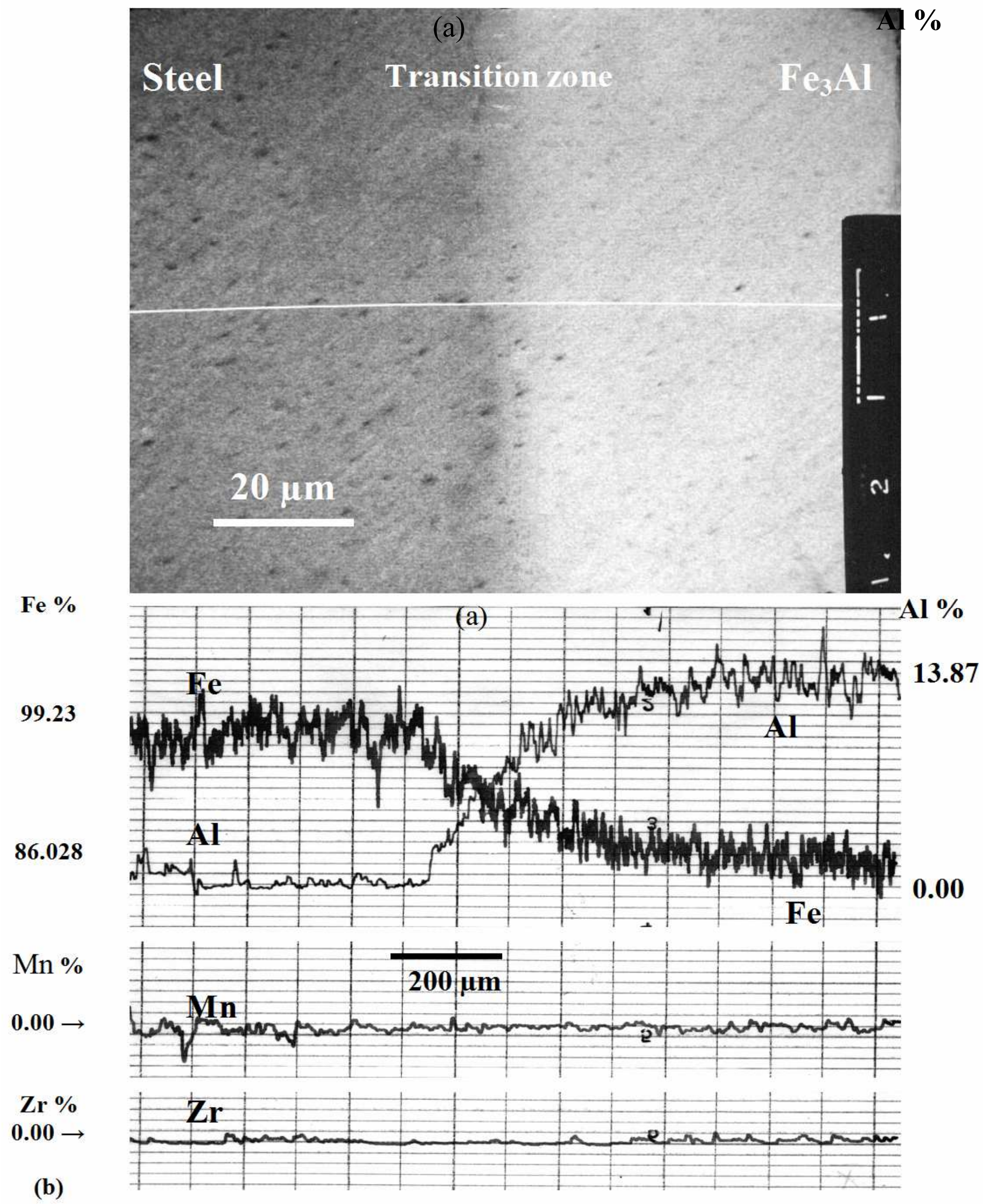


Figure 12: EPMA line analysis across the Fe_3Al/steel joint bonded at: T=1200 ^{0}C; t_b= 30 min. and P= 10 MPa: (a) View of the transition zone with line of microanalysis. (b) Distribution of Al, Fe, Mn and Zr elements across the transition zone.

X-ray diffraction

The phase constitutions formed in the Fe_3Al/steel joints were further researched by means of X-ray diffraction. The results of XRD investigation which were performed on the surfaces of samples fractured during shear testing on both sides. Diffraction standards shown in Figure (13) indicate the presence of aluminum iron carbide Fe_3AlC and Fe_3Al phases at the fracture surfaces of the Fe_3Al/steel joint bonded at 1100^{o}C; bonding time 60 min and bonding pressure 10 MPa.

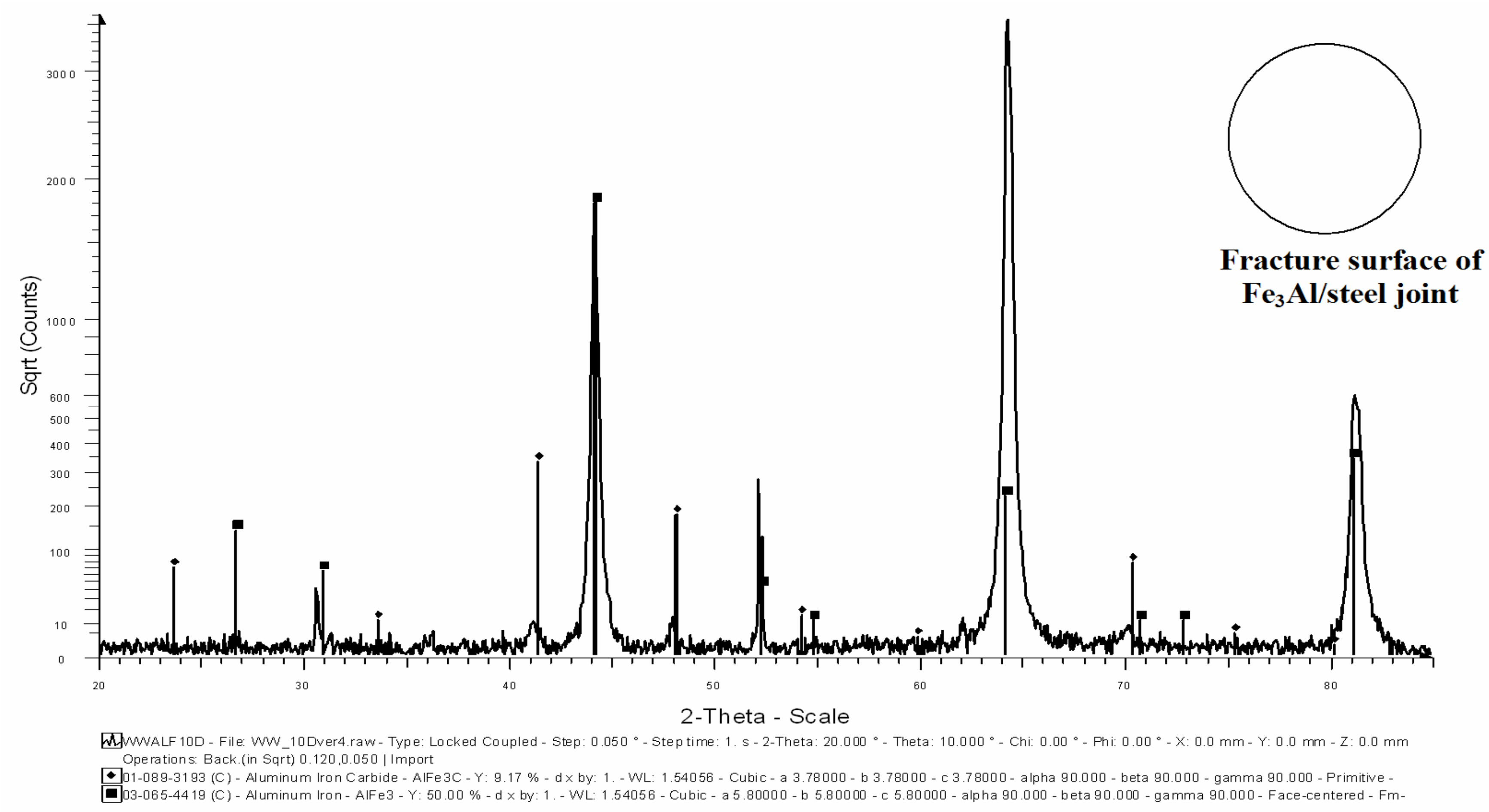


Figure 13: XRD pattern of the fracture surface of Fe_3Al/steel joint bonded at: T= 1100^oC; t_b=60 min and P= 10 MPa.

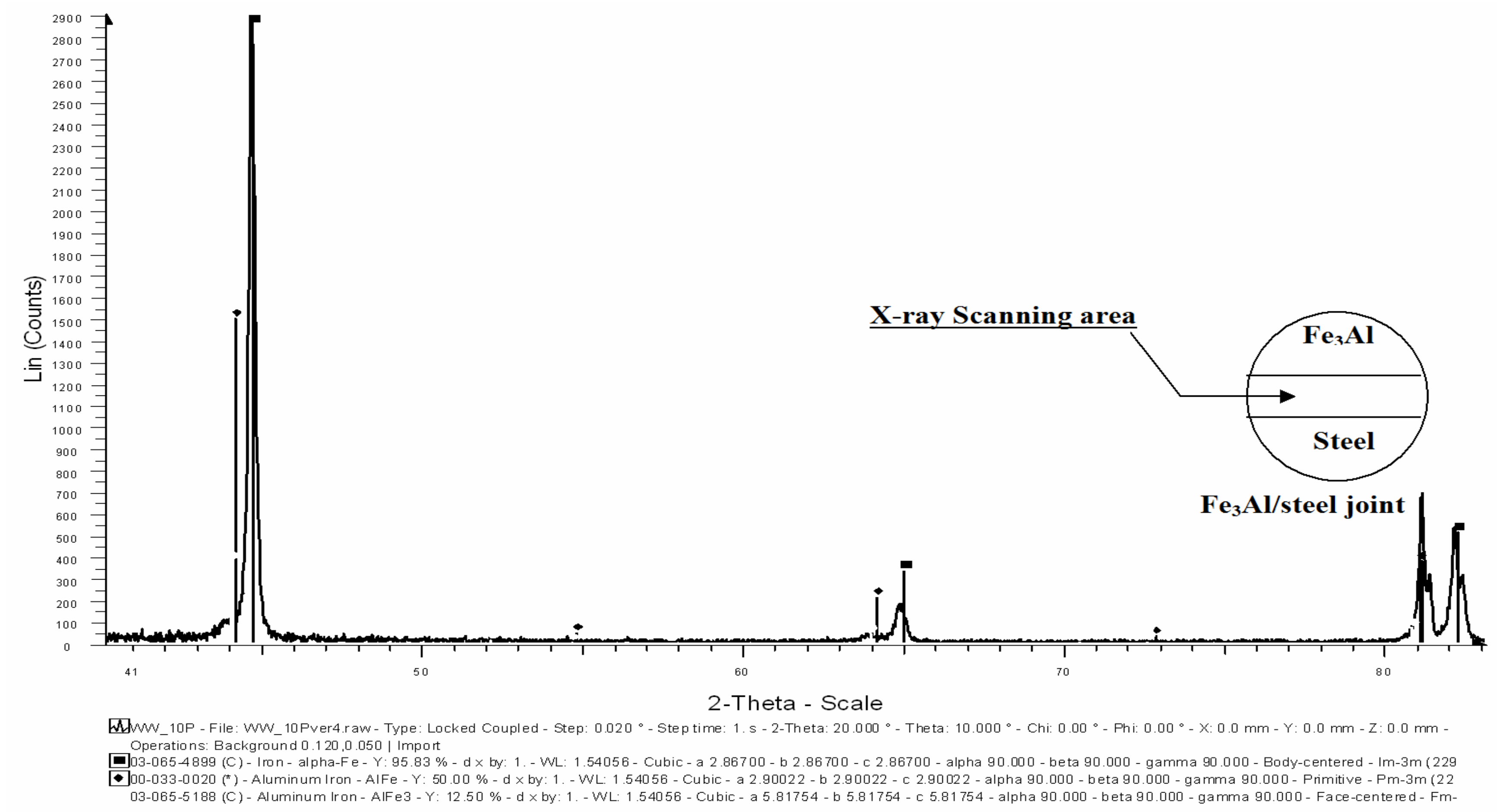


Figure 14: XRD pattern of the cross-section of Fe_3Al/steel joint bonded at: T= 1100^{o}C; t_b=60 min and P= 10 MPa.

Figure (14) shows the results of XRD investigation which were performed on the polished surfaces of samples which cut perpendicular to the joint transition zone. Diffraction standards in figure 14 indicate that there was α-Fe; FeAl and Fe_3Al phases present at the polished surface which cut perpendicular to the joint transition zone for Fe_3Al/steel joint bonded at 1100°C; for 60 min and bonding pressure of 10 MPa.
The results of the linear distribution indicated that the concentrations of Al and Fe were changed along the transition zone, it is also evident that Al and Fe diffused to the transition zone and reactions between these elements may occurred and these reactions was investigated by using X-ray diffraction and the results showed the existence of the new phase Aluminum iron carbide Fe_3AlC.

CONCLUSIONS

During experimental work of direct diffusion bonding of Fe_3Al to steel, the results obtained lead to the following conclusions:

- The maximum shear strength of Fe_3Al/steel joints was 362 MPa for joints bonded at 1100°C and for 60 min.
- The maximum width of the transition zone was 159 µm for joints bonded at bonding temperature of 1100°C and bonding time of 30 min.
- X-ray diffraction results showed the existence of new phases such as FeAl and Aluminum iron carbide Fe_3AlC.
- The transition zones in the middle between the Fe_3Al and the steel were clearly observed.
- The optimum conditions for joining Fe_3Al to steel joints are found to be at: $T=1100^oC$; $t_b=60$ min and P= 10 MPa.

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