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Solid state joining of steel-Cu P/M preform tubes: use of interlayer and post joining heat treatments

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Solid state transition joints made, by cold extrusion, between steel and Cu powder metallurgical (P/M) preform tubes were heat treated at various temperatures and time, with and without interlayer, to evaluate the weld strength and microstructural characteristics. Post-joining heat treatments lead to different recrystallized structures near the interface due to strain localization and solute drag effect. The strain localization and residual porosity in the joints enhanced void formation in the diffusion zone when the interlayer was absent. Coarse Ni powder as an interlayer reduced the weld strength due to physical discontinuities and residual particles at the interface, after heat treatment. On the other hand, finer Ni powder improved the weld strength. Use of 1 μm Ni interlayer in combination with optimal heat treatment conditions (950°C for 2 h) resulted in about 45% increase in weld strength.

1. Introduction

It is generally accepted that the strength of solid state joints depends primarily on the extent of surface expansion during deformation [1]. The surface expansion during P/M deformation would be relatively low when compared to their wrought equivalents; this is due to persistent densification of P/M preforms during deformation, which consumes part of the deformation energy and reduces the efficacy of deformation in causing the surface expansion. The low stretching at the interface of the two metals could lead to lower weld strength. Hence, the use of a post-joining heat treatment could be beneficial to increase the weld strength in the case of P/M processing. Post-joining heat treatment offers a possibility for improving the weld strength by causing inter diffusion and resultant metallurgical unification of metal pair, but could also result in certain adverse effects like micro-cracks, micro-voids and intermetallic compound formation depending on the affinity of metal pair with each other and their intrinsic diffusivities. The optimum heat treatment should improve the weld strength without causing these adversities.

The prior deformation was found to have significant effect on the diffusion kinetics of dissimilar wrought metal joints subjected to elevated temperature exposure [2]. It was shown that the diffusion of copper into iron increases sizably with prior deformation, but the rate of the diffusion increment drops with an increase in the temperature above the transformation line. These observations indicate the probability of greater diffusion mobility of copper into iron at a temperature range below the critical line, where the grain boundary and short circuiting diffusion provide high diffusion rate. Moreover, internal stresses at the interface and strain gradient in the bimetallic layers/components, which arise during simultaneous deformation, could also have an influence on the heat treated microstructure and weld strength of dissimilar metal joints.

Diffusional transformations between Armco iron and copper with different oxygen contents (ETP and OFLP) have been studied [3, 4]. The microstructure revealed iron oxide (wustite) at the interface in the joints made between ETP and Armco iron, suggesting the reaction of iron with oxygen dissolved in the ETP base metal. Some copper particles in the iron matrix, near the bond interface, also had been observed due to the solid-state precipitation of copper in iron and the eutectoid reaction ($\gamma \Rightarrow \varepsilon + \alpha$) at bonding temperatures around 900°C. This precipitation reaction increased the interfacial strength. High adhesion strength of steel-Cu bimetal sheet, made by cold cyclic rolling (70%) of copper powder, has been achieved by optimizing the sintering and final annealing temperature [5].

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The differential thermal expansion, interdiffusion and Kirkendall voids, which originate during heat treatment of dissimilar metal joints, could be eliminated by application of suitable interlayer between the two metals. The interlayer should be suitably reactive to both metals. Nickel was chosen as an interlayer in the present study due to its greater solubility for Fe and Cu, than vice versa condition [6]. Extensive efforts have been made in recent years to understand the formation and evolution of interdiffusion microstructures in wrought dissimilar joints made between Cu-304 SS, Ti alloy-SS, Cu-Ag, Cu-Ni and Al-Cu [7–12]. The interface microstructure and its effect on the performance of the diffusion bonds between microduplex stainless steel-Ti-6Al-4V, Ti alloys-SS and low alloys steels, Ti-Al, TiAl alloys to steel, etc. has also been studied [13–21]. The main objective of current investigation is to study the influence of post-joining heat treatment (with and without Ni powder interlayer) on the weld strength and interface microstructure of solid state joints between steel-Cu P/M preform tubes.

2. Experimental

The properties of steel and copper powders used in the present investigation are presented in Table I. The details of sintered P/M preform preparation from these powders could be seen elsewhere [22]. The process parameters for solid state joining of these materials, by cold extrusion, were optimized in earlier studies [22]. These process parameters and heat treatment conditions used in the present work are shown in Table II. Taguchi Method of design of experiments [23] was used to optimize the post-joining heat treatment cycle.

TABLE I Properties of the steel and copper powders used in the present investigation

Property	Steel powder		Copper powder	
Source	Hoganas India Ltd.		The Metal Powder Company Ltd.	
Composition (wt.%)	Fe: Balance		Cu: 99 min	
	C: 0.01			
	S: 0.008			
	H_2 Loss: 0.10			
Apparent density (g/cc)	2.95		2.74	
Flow rate, s/50 g	28		22	
Sieve analysis	μm	%	μm	%
	+212	0	+75	3 max
	–212+180	1.2	–45	45 min
	–180+150	8		
	–150+45	Bal		
	–45	15.4		

The two preforms were assembled by placing the low strength preform over the high strength preform as shown in Fig. 1a, followed by insertion of the mandrel with sleeve. The assembled P/M preforms were cold extruded using a double action hydraulic press of 1000 kN capacity. Experimental set-up and the sample arrangement are as shown in Fig. 1b. An attempt has been made to

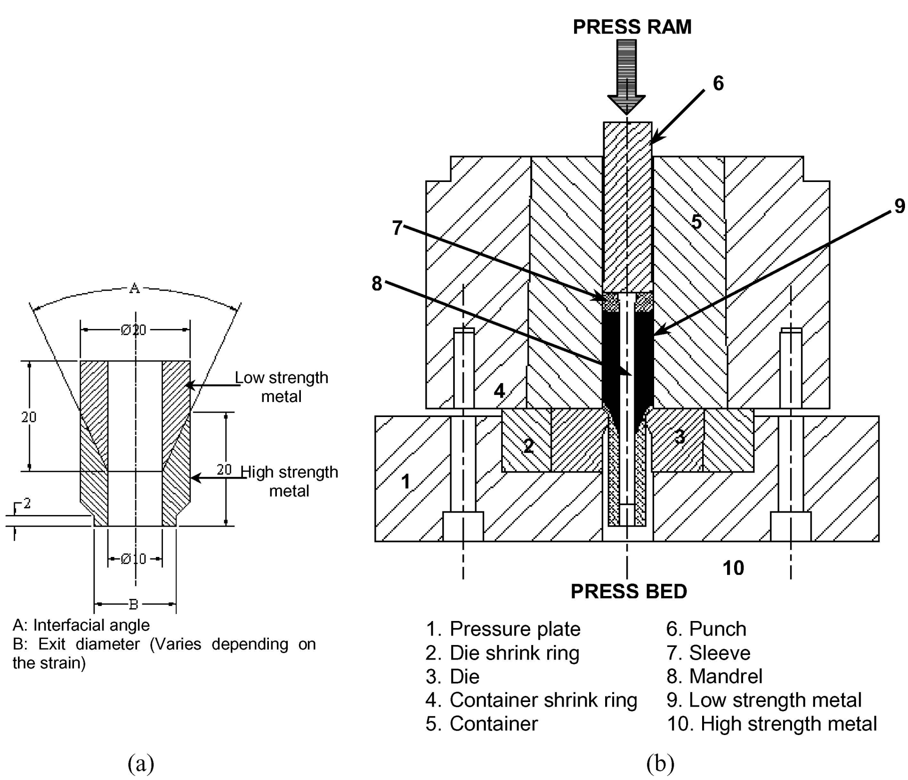


Figure 1 Schematic diagrams, (a) assembled preforms for the co-extrusion, (b) experimental set-up for co-extrusion.

TABLE II Extrusion parameters and heat treatment conditions used in the present investigation

Extrusion parameters	Heat treatment parameters			
	Interlayer	Particle size (μm)	Temperature (°C)	Time (h)
DR: 1.25	Ni	100	750	2
VR: 1.25		50	850[a]	4[a]
IA: 50°		1	950[a]	6[a]
ε: 1.00				

Remarks: DR-density ratio, VR-volume ratio, IA-interfacial angle, ε-strain.
[a]Heat treatment of the joints without interlayer was carried out at these conditions.

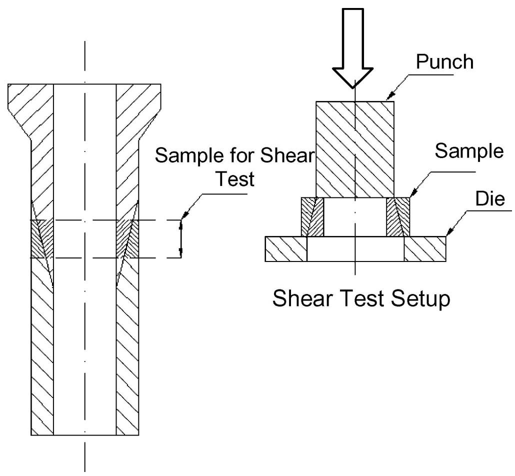


Figure 2 Selection of sample for weld strength evaluation and shear test setup employed in the present work.

avoid adverse effects like micro-voids and intermetallic compounds by using Ni interlayer. Excessive interaction of base metals with interlayer could lead to undesirable effects. Therefore, the Ni powder size was also varied. Nickel powder was mixed with carbon tetra chloride and applied to the surface of bottom preform. Then the preforms were assembled and extruded. These joints (with and without interlayer) were heat treated at different temperatures and time in semi-industrial pusher type electrical resistance furnace with N_2+H_2 (3:1) atmosphere.

The weld strength of the joints was determined by subjecting the bond line to a shear stress (Fig. 2). Since the joint configuration does not allow for the preparation of standard test specimen, shear test was used for evaluating the joint strength. Perhaps, it may not be out of place to add here that, use of shear test in evaluating the joint strength is quite common [24–31]. The sample for weld strength evaluation was taken from the center of interface region (Fig. 2). During shear testing (to evaluate the weld strength of the joints) the stress required to separate low strength preform from high strength preform were measured in terms of the load required to eject the softer preform from the joined P/M preform tube. The interfacial area was measured and force recorded. Then the weld strength was evaluated. The samples for metallographic examination were appropriately sectioned, mounted, mechanically polished and then etched with 5 g copper sulphate, 2 ml HCl and methanol solution for copper and with 5% nital solution for steel. The microstructural features at the bond region were examined by using optical and scanning electron microscope (SEM) and energy dispersive x-ray micro analysis (EDX). Supportive inferences were obtained through microhardness measurements (load 100 g) across the joints.

3. Results and discussion

3.1. Heat treatment without interlayer

Typical microstructures of as-sintered P/M preforms are shown in Fig. 3. The shear test results of heat treated joints made without interlayer are furnished in Table III. The weld strength was found to increase with heat treatment temperature and time, as shown in Fig. 4. The effect of heat treatment temperature was always stronger than time, as the temperature decides the driving force for any reaction. Heat treatment at high temperatures decreases the time required for the elimination of original interface and therefore resulted in higher weld strength. Besides, at these temperatures the grain growth is an important

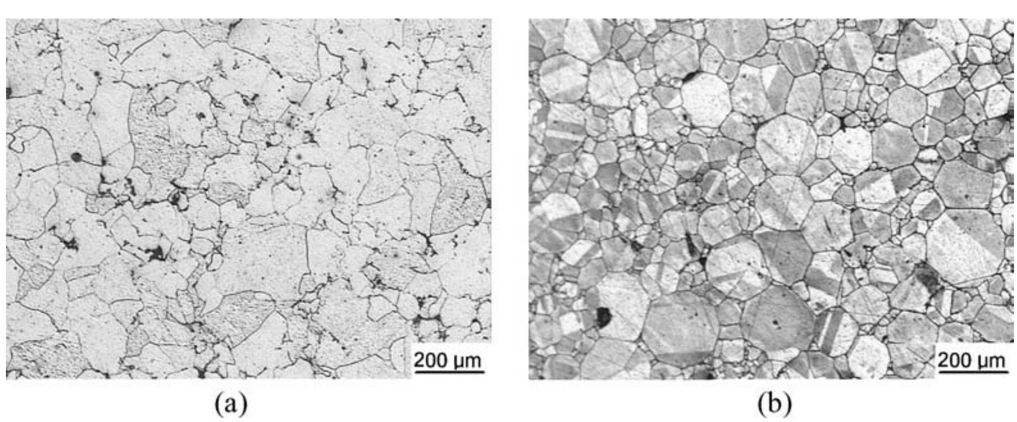


Figure 3 Typical etched microstructure of the sintered P/M preforms in as sintered condition, (a) Steel, $\rho = 93\%$, etched with 5% nital (b) Copper, $\rho = 86\%$, etched with 5 g copper sulphate, 2 ml HCl and methanol solution.

TABLE III Influence of heat treatment parameters on the weld strength (MPa) of joints made without interlayer

Exp. details	Sample-1	Sample-2
1–850°C, 4 h	45	45
2–850°C, 6 h	34	36
3–950°C, 4 h	43	47
4–950°C, 6 h	87	89
As extruded	60	

Remarks: The as extruded weld strength values were taken from [22].

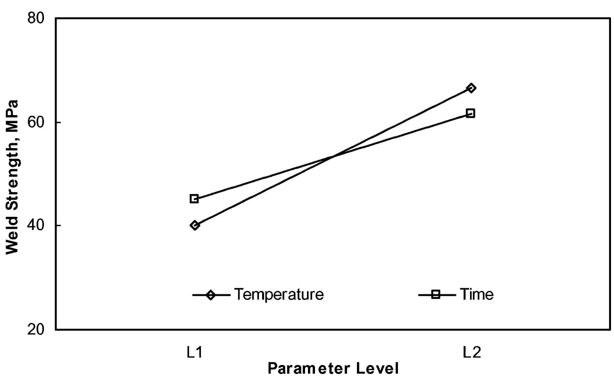


Figure 4 Influence of heat treatment temperature and time on the weld strength of steel-Cu joints made without interlayer [L1, L2 = 850°C, 950°C for heat treatment temperature and 4 h, 6 h for heat treatment time].

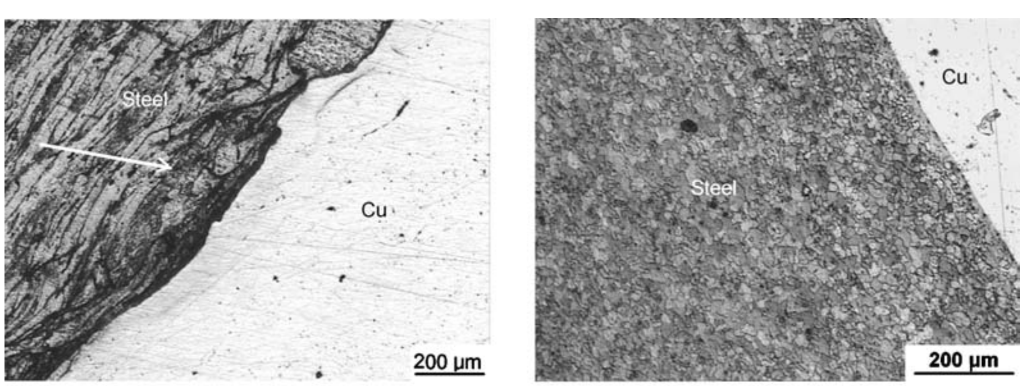
process in the removal of the initial bond interface leading to full bonding and high weld strength [32]. The results show that a heat treatment cycle of 950°C, 6 h would give highest weld strength of 75 MPa, which is 25% higher than the strength of these joints in as-extruded condition (60 MPa). During shear testing all the samples failed at the interface as the top punch suits the diameter of the inner softer ring. Therefore, the failure location did not change with the heat treatment conditions.

3.1.1. Influence of strain localization and diffusion

During solid state joining of dissimilar P/M preforms by cold extrusion the deformation at or near the interface will be higher than at the regions away from it [22], as a result of high interfacial friction. The strain localization could lead to high dislocation density at or near the interface, which would quickly reach to a limiting value. The band of highly localized deformation near the interface would have high stored energy and provide greater driving force for the recrystallization during subsequent heat treatment. Interface microstructures in as-extruded condition revealed severely deformed grains on steel side of the interface (Fig. 5a). This factor resulted in variation of recrystallized structure from interface to the regions away from it. The microstructural variation can be seen from Fig. 5b, where the grain size was smaller near the interface (~12–15 μm) than in the regions away (~33–36 μm) from the interface. The localized strain may also affect the diffusion kinetics during heat treatment due to higher diffusivities of cold worked metals than annealed or defect-free equivalent metals. The cold extrusion increases the defect density (crystal imperfections) in metals and thus these crystal imperfections would have aided in the diffusion process.

Qualitatively, grain size near the interface varies depending on the solute drag effect and the stress state in the diffusion region and resultant plastic deformation. The solute additions, even in small amounts, could lead to sharp decrease in grain boundary mobility [33]. In the present work, during heating to soaking temperature, diffusion of metal atoms would have taken place across the interface before the initiation of recrystallization of deformed metals. Besides, the strain localization near the interface enhances the diffusion. Therefore, it is reasonable to expect that the diffused (along the grain boundary and dislocation cell walls) metal species act as pinning points to the movement of grain boundary during recrystallization leading to relatively smaller grains near the interface.

Moreover, the solute drag was considered to be more effective when the solubility of the solute in the metal is limited. The steel-Cu system satisfies the above condition, i.e., low solubility of 'Cu' in 'Fe'. In general, the presence of minor solute concentration (in the present



(a) (b)

Figure 5 Strain localization (arrows) and resultant microstructure of solid state joints of P/M preforms, etchant: 5% nital (a) optical microstructure before heat treatment (as-extruded), (b) after heat treatment (850°C, 4 h).

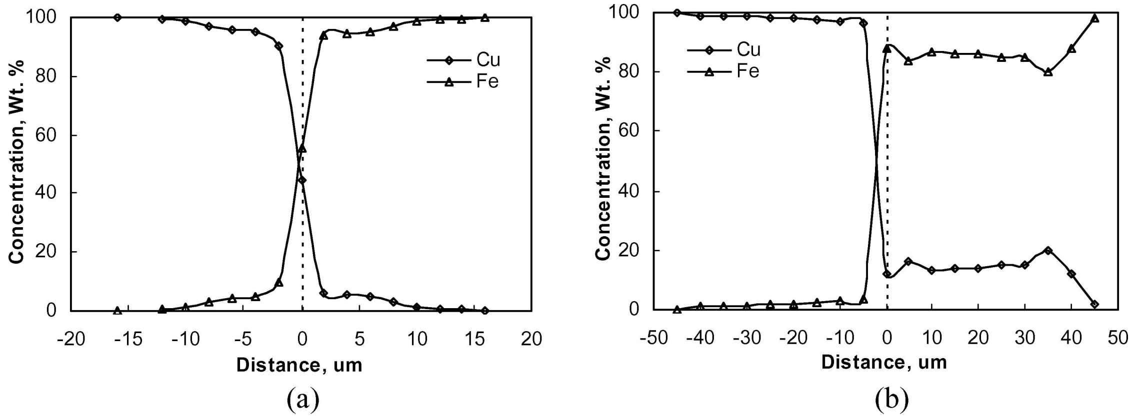


Figure 6 The concentration profiles of Cu and Fe at the interface of steel-Cu joints, (a) joint heat treated at 850°C for 4 h, (b) joint heat treated at 950°C for 6 h.

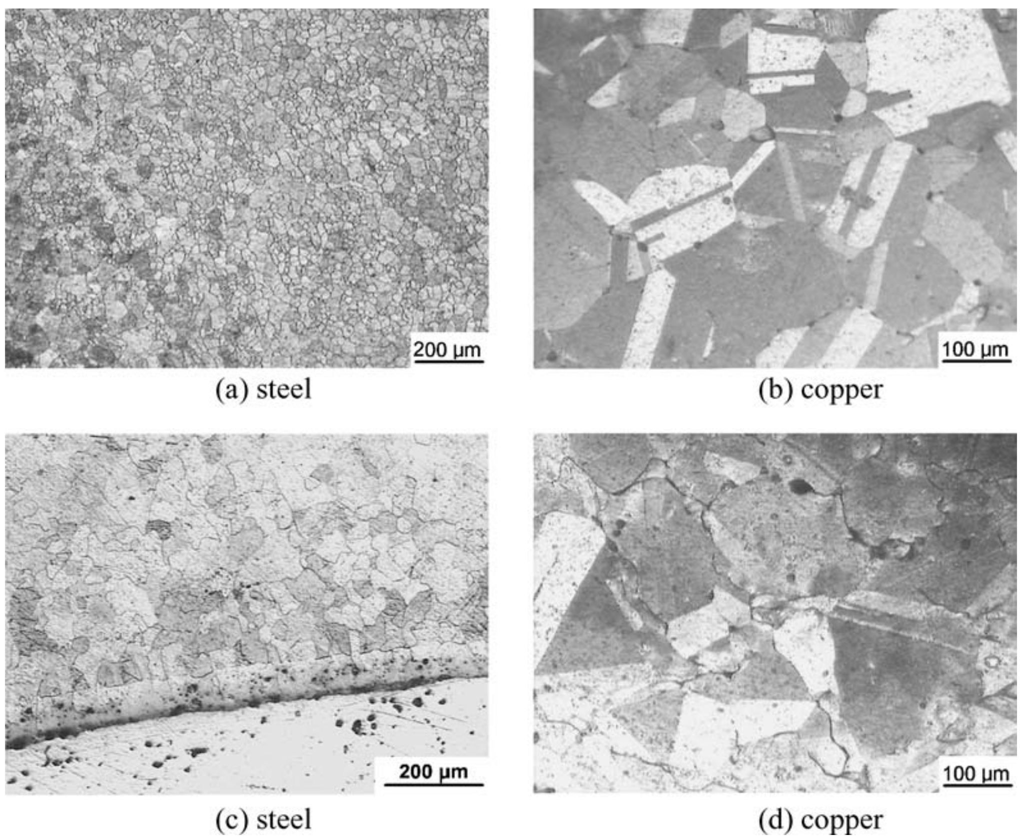


Figure 7 Optical microstructures after heat treatment, etchant: 5% nital for steel and 5 g copper sulphate, 2 ml HCl and methanol solution for copper (a) and (b) at 850°C, 4 h, (c) and (d) at 950°C, 6 h.

case 'Cu in steel or Fe in Cu') lead to increase in the activation energy of recrystallization and in turn the recrystallization temperature [34]. This supposes that the regions with high 'Cu' concentration (Fig. 6) near the interface (on steel side) would have high recrystallization temperature. Therefore, for a given temperature and time the grain growth would be low at the interface than at regions away from it (Fig. 5b). Similar trend could be expected in the copper side of the joint. However, some grain growth seemed to have occurred (especially in the regions away from the interface), as the heat treatment temperature was relatively higher than the recrystallisation temperature of pure copper (Figs. 3b and 7b and d). At high heat treatment temperatures the drag effect was observed to be negligible leading to grain growth (Fig. 8). This is attributed to the reduced segregation of diffusing species along the grain boundaries. At high temperatures the segregated elements diffuse to the grain interior and tend to homogenize the region, which could not exert the drag effect on growing/moving grain boundaries resulting in grain growth (Fig. 8). However, the weld strength at this condition was found to be high due to reduction in

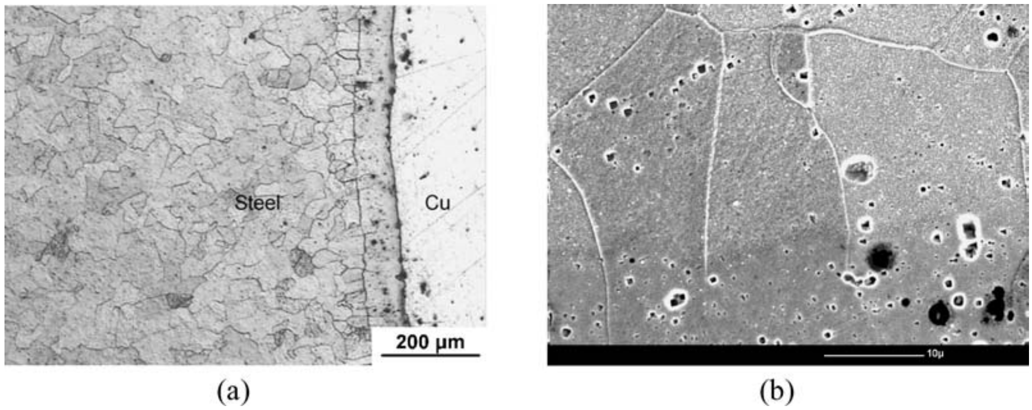


Figure 8 Microstructures near the interface of steel-Cu joints, etchant: 5% nital (a) etched optical microstructure of the joint heat treated at 950°C for 6 h, (b) SEM micrograph of the joint heat treated at 950°C for 6 h.

the abrupt nature of the interface (as observed in the as-extruded condition, Fig. 5a) resulting in smoother transfer of load between the metal pair.

3.1.2. Diffusion induced micro-voids

Another phenomenon that can occurs during the inter-diffusion of two dissimilar metal atoms across the common interface is the stress build-up in the diffusion zone. The region, which loses mass undergo tensile stress and the one that gains mass would be under compression. During heat treatment the stress fields build-up and result in plastic flow in the diffusion region. The tensile stresses could contribute to the development of voids in the region of maximum mass loss [7–12]. Microstructural examination at the bond region of steel-Cu joints showed large number of micro-voids on steel side (Fig. 9). The diffusion coefficient of iron in pure copper was observed to be approximately one order of magnitude higher than copper in pure iron [35]. This difference suggests that the void formation would occur in the steel, next to the interface.

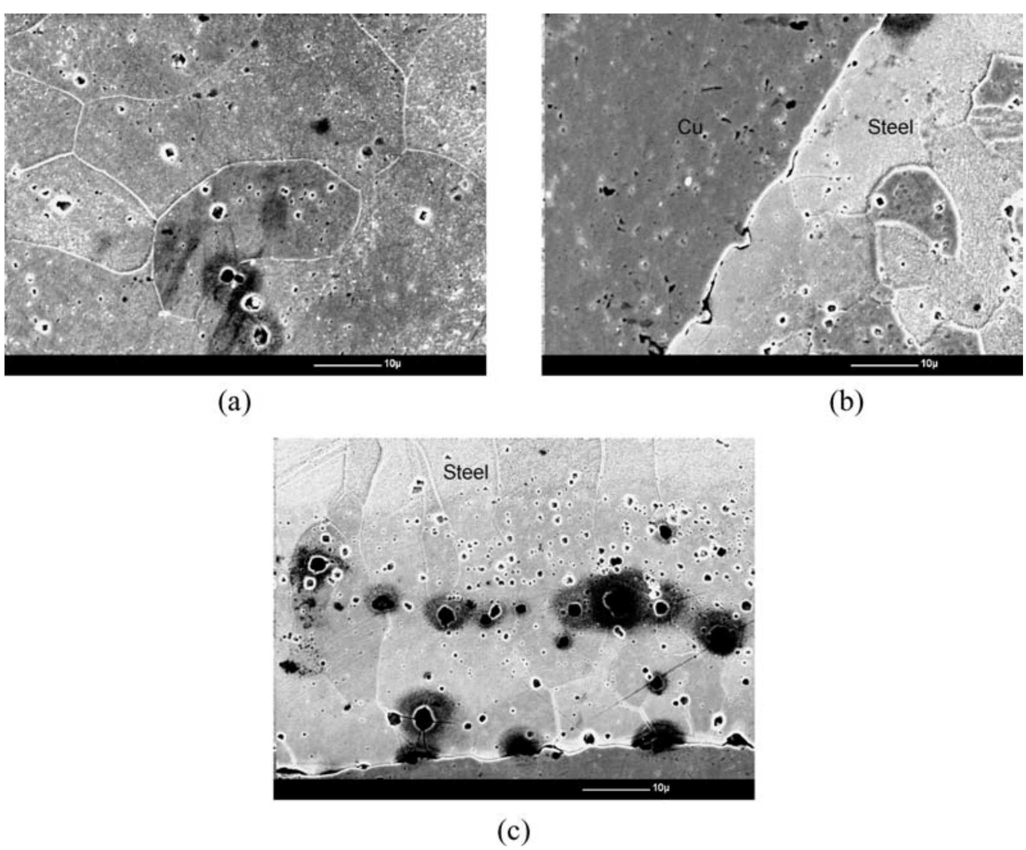


Figure 9 SEM microstructures near the bond region of steel-Cu joints showing the diffusion induced voids/porosity, etchant: 5% nital (a) microstructure of the bulk, (b) joint heat treated at 850°C for 4 h, (c) joint treated at 950°C for 6 h.

Pores in the diffusion zone (Fig. 9b and c) generally form due to heterogeneous nucleation of vacancies produced by unequal diffusion flow of atoms across the interface. The nuclei could be dislocations [36], grain boundaries [36, 37], small voids [38], or inclusions [38–41]. In the present work the high dislocation density at the interface (due to strain localization during cold extrusion) and the presence of residual porosity (since the joints were made between P/M preforms) would have facilitated the pore formation by acting as favourable nucleation sites. The tensile stress that exists in the region of the diffusion zone was also a contributing factor in the development of these voids.

Figure 9c shows that the pores are widely distributed (over 30–40 μm from the interface). It was found that the influence of diffusion-induced porosity on the weld strength was very low. These pores would not have influenced the initiation of cracks at the interface during 'shear test' methodology used to evaluate the strength. In shear testing the stress-state would be compressive in nature at the tool-metal interface, which is incapable of accentuating the crack initiation in the presence of pores. Besides, the pores are present on steel side, whereas in shear test the load was directly applied on the copper preform (Fig. 2). Nevertheless, the presence of these micro voids might pose problems in long time stability and reliability of these joints. Hence a need arises to eliminate these voids.

The concentration profile of Cu and Fe at the interface of steel-Cu joints heat treated at two different conditions is presented in Fig. 6. It was observed that the concentration profiles cross each other in the copper region. However, these concentration profiles should have crossed each other at the interface, but their intersection was detected ~2 μm inside the Cu region, for the joints heat treated at 850°C for 4 h and ~5 μm for the joints heat treated at 950°C for 6 h. This apparent discrepancy is attributable to Kirkendall effect. The thickness of the region with high Cu concentration, on steel side, was found to increase with heat treatment temperature and also with time. The thickness was around 10 μm for the joints heat treated at 850°C for 4 h and it was grown to ~40 μm for the joints treated at 950°C for 6 h (Fig. 6).

The microhardness variation across the interface is shown in Fig. 10. The hardness near the interface, towards steel side, increased due to diffusion of copper into steel. The narrow zone of high hardness was found to increase with temperature. The copper atoms either dissolve in α-Fe or precipitate as ε-Cu particles without forming intermetallic compounds with Fe. The XRD plot of diffusion region of steel did not show the copper peaks (Fig. 11). This confirmed that all copper was dissolved in steel. Moreover, substantial shift in the position of iron XRD peaks was observed with respect to the pure iron peaks. These shifts suggest the expansion in the iron lattice parameter. Similarly the Cu dissolution in iron resulted in expansion of the iron lattice parameter in an earlier investigation [42] and the extent of expansion increased with concentration of copper in steel (<8%). This suggests that by dissolution of copper atoms in Fe the iron lattices expands and results in high local hardness. The hardness of copper seems to be unaffected by the heat treatment, except slight increase near the interface.

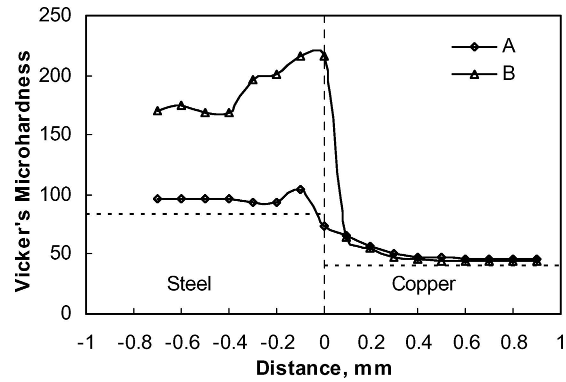


Figure 10 Hardness profile across the steel-Cu joints made without interlayer, A: 850°C, 4 h, B: 950°C, 6 h (dotted line represents the hardness in as-sintered condition).

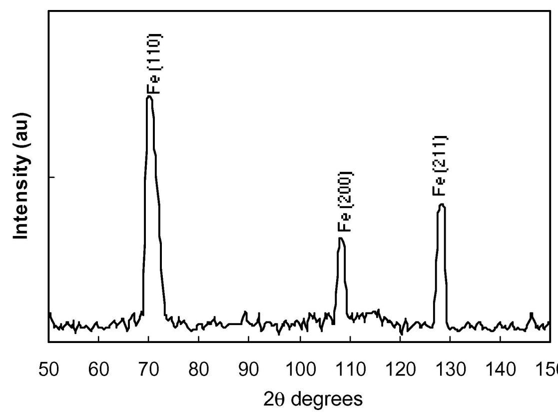


Figure 11 XRD plot of diffusion zone towards steel in steel-Cu transition joints heat treated at 950°C for 6 h.

3.2. Heat treatment with interlayer

The strength of heat treated joints made with Ni powder interlayer is presented in Table IV. Influence of heat

TABLE IV Weld strength (MPa) of heat treated joints made with interlayers

Exp. no	Process details	Sample-1	Sample-2
1	Ni, 100 μm, 750°C, 2 h	57	55
2	Ni, 100 μm, 850°C, 4 h	52	54
3	Ni, 100 μm, 950°C, 6 h	59	63
4	Ni, 50 μm, 750°C, 6 h	47	49
5	Ni, 50 μm, 850°C, 2 h	55	55
6	Ni, 50 μm, 950°C, 4 h	57	55
7	Ni, 1 μm, 750°C, 4 h	52	54
8	Ni, 1 μm, 850°C, 6 h	78	76
9	Ni, 1 μm, 950°C, 2 h	86	89

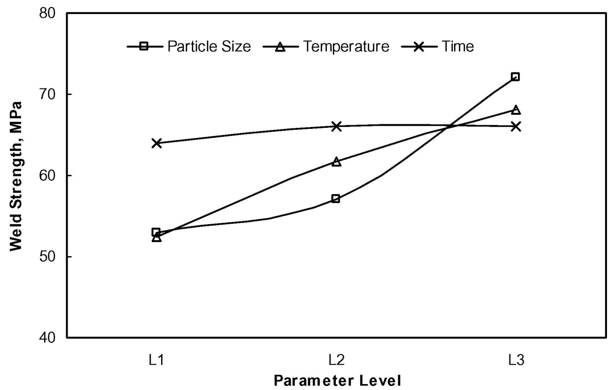


Figure 12 Influence of process parameters on the weld strength of heat treated steel-Cu joints made with Ni interlayer [L1, L2, L3 = 750°C, 850°C, 950°C for heat treatment temperature, 2 h, 4 h, 6 h for heat treatment time and 100 μm, 50 μm, 1 μm for Ni powder interlayer particle size].

treatment parameters on the weld strength is shown in Fig. 12. Highest weld strength was observed with 1 μm Ni powder interlayer and highest heat treatment temperature. The heat treatment time had little influence on the weld strength. The results revealed that heat treatment temperature have strong influence on weld strength followed by interlayer particle size and then the heat treatment time. Highest strength of 87 ± 8 MPa was observed in these joints when heat treated at 950°C for 2 h with 1 μm Ni powder as an interlayer. The post-joining heat treatment with interlayer resulted in 45% increase in the weld strength when compared to the strength in the as-extruded condition (60 MPa).

3.2.1. Influence of interlayer particle size

As-polished microstructures of different joints are shown in Fig. 13. Some physical discontinuities were observed at the interface (Fig. 13a) when the interlayer particles were coarse. As the particle size reduced to 1 μm these discontinuities were found to diminish (Fig. 13c). Possible reason can be that for the same normal and shear pressure at the interface (since the extrusion process parameters are constant), coarser particles at the interface would not have allowed the two metals to come into intimate contact over the entire surface of the particle. Besides, the local strain hardening of the metal pair increases the stress required for further deformation of the metal to cover the whole surface of the coarse interlayer particles. These attributes left a small discontinuity at the interface near the edges (whose tangent is normal to the interface) of the coarse interlayer particles. As the particle size was reduced, these discontinuities also reduced in size, which were subsequently eliminated during heat treatment. In case of joints made with coarse interlayer particles these voids reduced in size (after heat

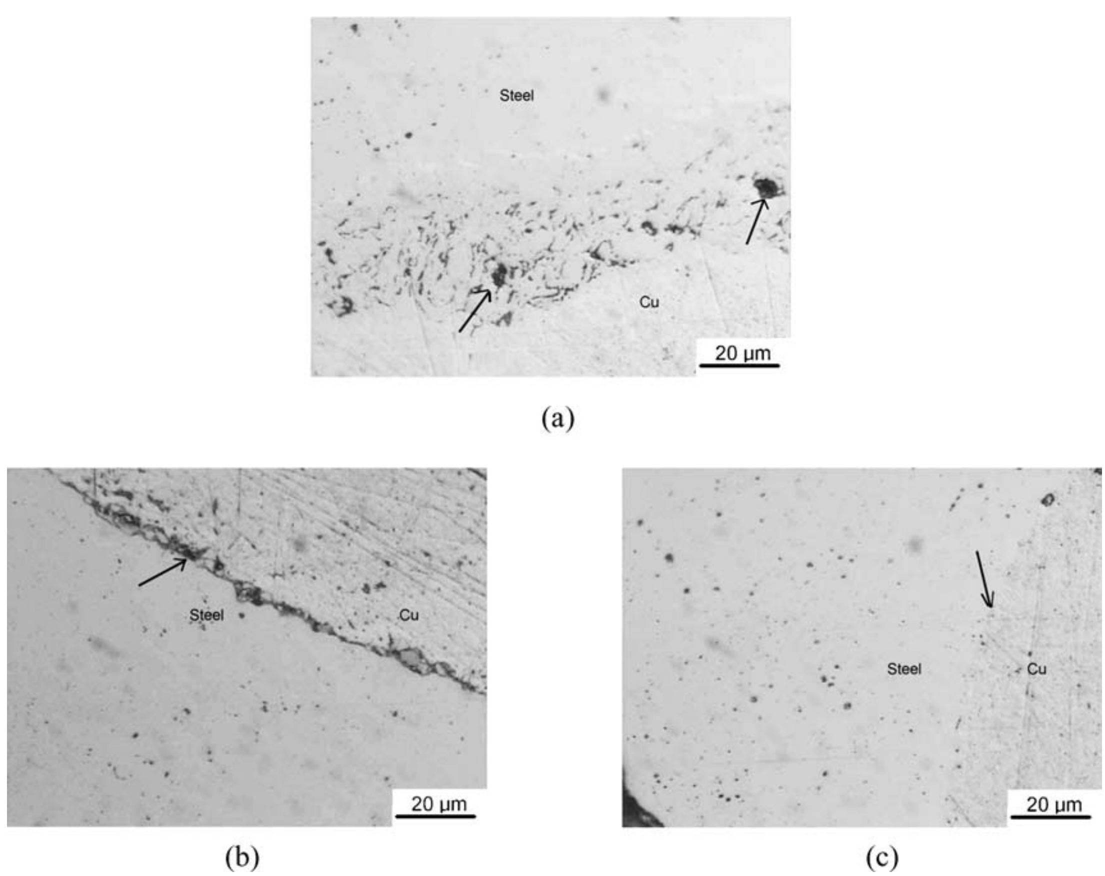


Figure 13 As-polished optical microstructures of steel-cu joints made with Ni powder interlayer and heat treated at 850°C for 4 h (a) 100 μm particle size, (b) 50 μm particle size, (c) 1 μm particle size.

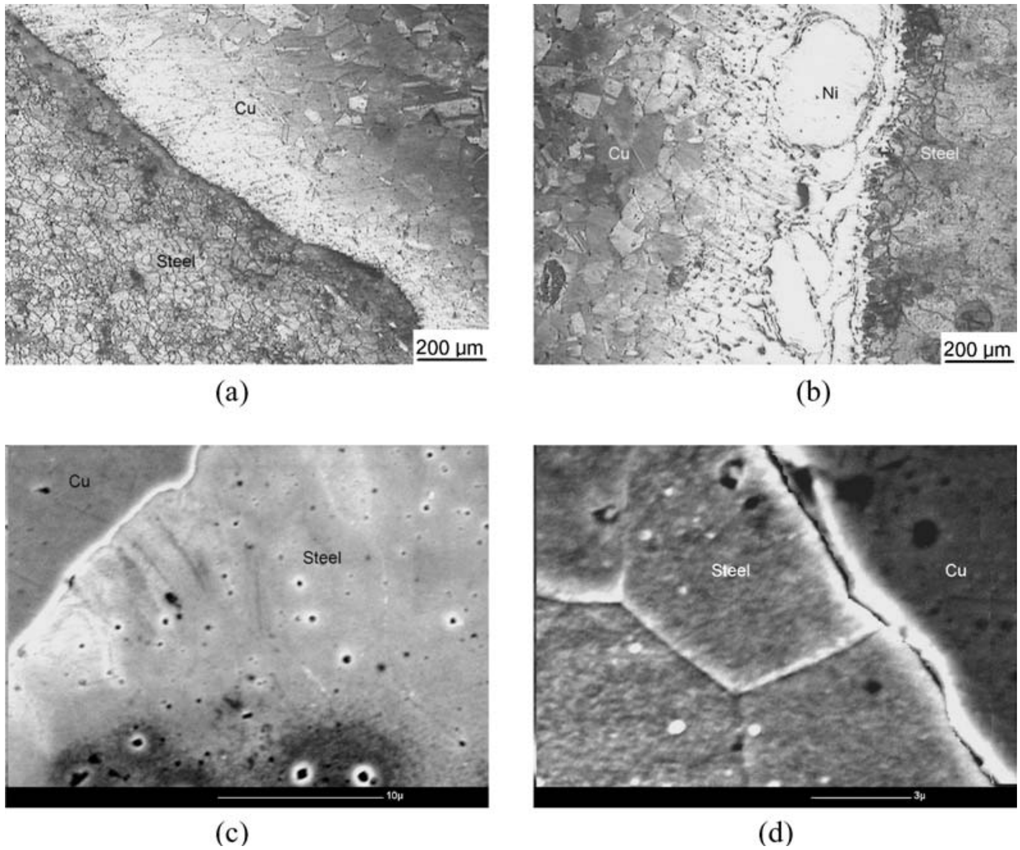


Figure 14 Interface microstructure of steel-Cu joints made with Ni powder interlayer, etchant: 5% nital for steel and 5 g copper sulphate, 2 ml HCl and methanol solution for copper (a) optical microstructure, 1 μm, 950°C, 4 h, (b) optical microstructure, 100 μm, 850°C, 4 h, note the residual interlayer particles (c) SEM micrograph, 100 μm, 950°C, 6 h, note the Kirkendall voids, (d) SEM micrograph, 1 μm, 950°C, 4 h.

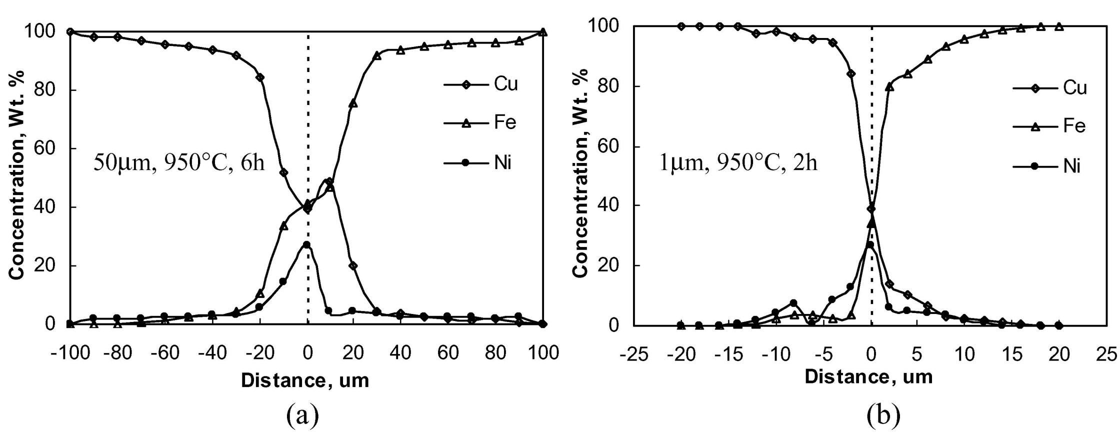


Figure 15 Concentration profiles at the interface of steel-Cu joints made with Ni as interlayer.

treatment) but total elimination was absent. This is one of the reasons for lower weld strength of joints made with coarse interlayer particle size (100 μm), Fig. 12.

Etching of these heat treated joints revealed different microstructures depending on the interlayer particle size, temperature and time. The uniformity of microstructure increased with decrease in the interlayer particle size (Fig. 14). More residual interlayer (after heat treatment) powder was found in the joints made with coarse interlayer particles, which along with the physical discontinuities at the interface reduced the weld strength (Fig. 12).

3.2.2. Influence of interlayer

In the steel-Cu joints made with Ni interlayer the diffusion of Ni into the steel was found to be less than into the copper (Fig. 15). This is because the diffusivity of Ni in steel ($\sim$2.0 $\times$ 10^{-16} cm^2 s^{-1}) is less compared to its diffusivity in copper, $\sim$2.70 $\times$ 10^{-10} cm^2 s^{-1} [43]. The concentration profile also showed that the depth of Ni diffusion (in steel and copper) reduces with reduction in interlayer particle size. The concentration profile revealed that the Ni interlayer could not completely suppress the inter-diffusion of steel and copper (Fig. 15). In the present

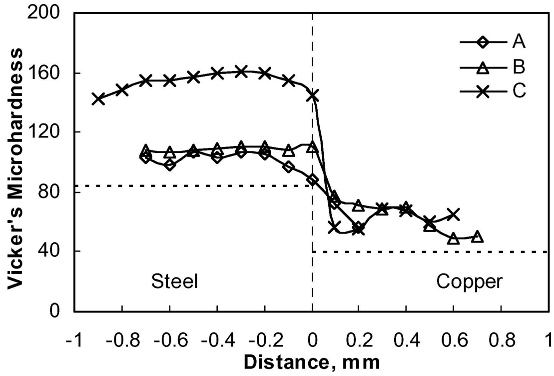


Figure 16 Micro hardness variation across the interface of joints made with Ni interlayer and heat treated at 850°C for 4 h (A) 1 μm particle size, (B) 50 μm particle size, (C) 100 μm particle size (dotted line represents the hardness in as-sintered condition).

work the interlayer was applied in the form of powder and the powder would not have covered the entire bond interface without leaving some gaps. These gaps could have acted as paths for diffusion of metal pairs into each other. Finer particles facilitated more uniform coverage of the bonding surface and consequent reduction in inter-diffusion. This minor inter-diffusion was observed to have no harmful effects on either microstructure or the weld strength, especially when the interlayer particles are fine. In the case of coarser particles, some Kirkendall voids were observed due to excessive interaction of base metals with Ni interlayer (Fig. 14c).

The microhardness variation across the heat treated joints is presented in Fig. 16. The high hardness (compared to the hardness in heat treated joints made without interlayer) on steel side was thought to be due to solid solution strengthening (Cu and Ni diffusion). Similarly the copper hardness was also found to be high (Fig. 16). Though the hardness of steel (near the interface) was more in the joints made with coarse interlayer particles (due to greater diffusion of Ni) the weld strength was found to be low due to physical discontinuities at the interface. In addition, coarse particles resulted in fine Kirkendall voids on steel side (Fig. 14c). On the other hand, uniform microstructure was obtained with finer Ni powder interlayer (Fig. 14a). Finally the microstructures of the base metals (far away from the interface) were not deteriorated due to post-joining heat treatment. It can be expected that the grain growth would be very high under the present experimental conditions of holding for longer time at elevated temperature. However, it was observed that there was no noticeable grain growth in base metals. This could be due to the thin oxide layer present on the powder particles, which hindered the recrystallization and minimized the grain growth. The optimal heat treatment conditions without deleterious effects in terms of excessive grain growth, interdiffusion, Kirkendall voids, etc., with highest weld strength were found to be 1 μm Ni powder and a heat treatment cycle of 950°C for 2 h.

4. Conclusions

The post-joining heat treatment of transition joints between steel and Cu P/M tubular preforms was carried out with and without interlayer. From the weld strength and microstructural observations the following conclusions were drawn.

- The strain localization near the interface lead to different recrystallized structure after heat treatment. 'Solute drag effect' resulted in finer grains near the interface.
- The strain localization and the residual porosity in the joints enhanced the void formation near the interface, in the absence of interlayer.
- Finer Ni powder as an interlayer facilitated in achieving highest weld strength. While the coarse powder reduced the weld strength due to physical discontinuities and residual particles at the interface.
- The optimal heat treatment conditions with 45% increase in the weld strength were observed to be Ni powder interlayer with 1 μm size and a heat treatment cycle of 950°C for 2 h.

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