CHAPTER 3

3 EXPERIMENTATION

The objective of this work was to clad a 3 mm-thick layer of copper onto a mild steel substrate to make its surface corrosion resistant, thus modifying the surface properties of the steel substrate. This study consists of extensive experiments along with a detailed analysis of the obtained results. In this chapter, the entire experimental procedure of cladding through FSW has been discussed, along with the method of obtaining samples for characterization from the cladded plates and the preparation of samples for characterization as per the established standards. In this work, the existing FSW setup was used in such a way that cladding was made possible with an optimised tool pin offset distance between the adjacent passes to effectively clad the plate in the minimum time. The cladded plates were characterised using various characterization techniques, including microstructural examination, mechanical testing like tensile testing, fractography analysis, microhardness evaluation and bend testing. A corrosion rate comparison was carried out to evaluate the behaviour of the copper surface before and after cladding with respect to base copper and steel. The surface topography of the corroded samples has been evaluated through atomic force microscopy.

3.1 Work material selection

AISI 1018-grade mild steel has been chosen as the substrate material in this study. Mild steel belongs to the category of steel having a low carbon content, with the carbon range falling below 0.25%. Global steel production in 2022 reached 1,878 million metric tonnes, with a 53.9% share of China and a 6.6% share of India, with 124.8 million metric tonnes [195]. AISI 1018 steel is a popular material that is used for numerous industrial applications and for making a variety of components like gears, axles, sprocket assemblies, shafts, and machinery parts for automotive engines due to its high tensile strength and good formability properties. AISI 1018 carbon steel is a free-machining grade that is the most commonly available grade around the world [196]. The only major issue with this steel material is its low corrosion resistance. A popular material that can be used to improve the corrosion resistance of steel is cladding the steel with copper. Copper offers high corrosion resistance and, thus, can act as a barrier against the corrosion of 1018 mild steel. Apart from these, copper, when cladded with steel, also improves the current-carrying capacity of steel and can have a potential application for bus bars. Therefore, the clad material chosen for this study is copper. Because of the obvious advantages of thick cladding, as discussed in section 1.6, it was decided to deposit a 3 mm-thick copper sheet over a mild steel substrate. The dimensions of steel plate and copper sheet were 200 mm x 70 mm x 6 mm and 200 mm x 70 mm x 3 mm, respectively, and are shown in Figure 3-1, and their elemental compositions are shown in Table 1. The initial trial experiments for cladding were carried out on an aluminium substrate with dimensions of 200 mm x 70 mm x 6 mm. The mill report was provided by the supplier; however, the received materials were again tested by optical emission spectroscopy to confirm the chemical compositions.

Table 1: Elemental composition of plates used for experimentation

Element	C %	Si%	Mn%	Cr%	Ni%	Fe%
Wt.%	0.170	0.010	0.995	0.089	0.018	Bal.
Element	**Zn %**	**Pb%**	**P%**	**Fe%**	**S%**	**Cu%**
Wt.%	2.354	0.055	0.045	0.039	0.047	Bal.
Element	**Cu%**	**Mg%**	**Mn%**	**Ti%**	**Fe%**	**Al%**
Wt.%	0.251	0.086	0.062	0.038	0.083	Bal.

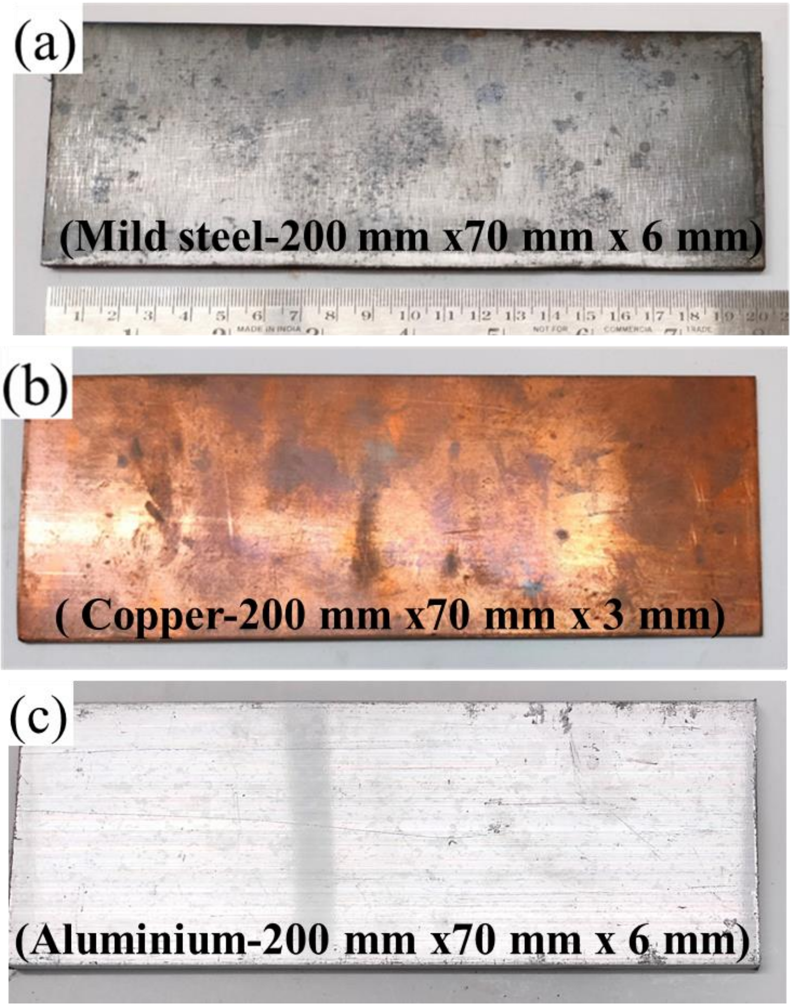

Figure 3-1 Work material used (a) mild steel (b) copper (c) aluminium

3.2 Friction stir welding machine

The entire cladding experiment was performed on a 3-tonne axial load (maximum thrust in the vertical direction that can be applied through the machine) numerically controlled FSW machine, which is shown in Figure 3-2. This machine has the capability to weld both ferrous and non-ferrous alloys. The entire machine setup has been shown in Figure 3-2b while the enlarged view of the main components is shown in the remaining figures. The WC tool was inserted in the tool holder, which rotated at the RPM at which the machine spindle was rotating and has been shown in Figure 3-2a. The control switch panel has been shown in Figure 3-2c through which the machine spindle was provided motion manually in upward or downward directions, and the table was given motion manually in positive and negative X directions to bring the specific location of the table beneath the tool pin. The welding/cladding process setting screen to feed the input parameters and the status monitoring screen to monitor the ongoing operation have been shown in Figure 3-2d and Figure 3-2e respectively. The plate holding arrangement with the plates clamped on the table has been shown in Figure 3-2f. Copper was placed on the top of the mild steel substrate and secured by the clamps, restricting all possible degrees of freedom of the plates. The machine is capable of cladding up to 300 mm in a linear direction. The machine has a numerical control for movement along the X and Z directions. The Y-axis movement is used in order to place the welding tool just above the plates' adjoining edges, manually. The desired tool tilt could be provided to the tool spindle in the range of ±5°. The machine could be operated in position control mode or load control mode; however, the entire cladding experimentation was performed in position control mode only.

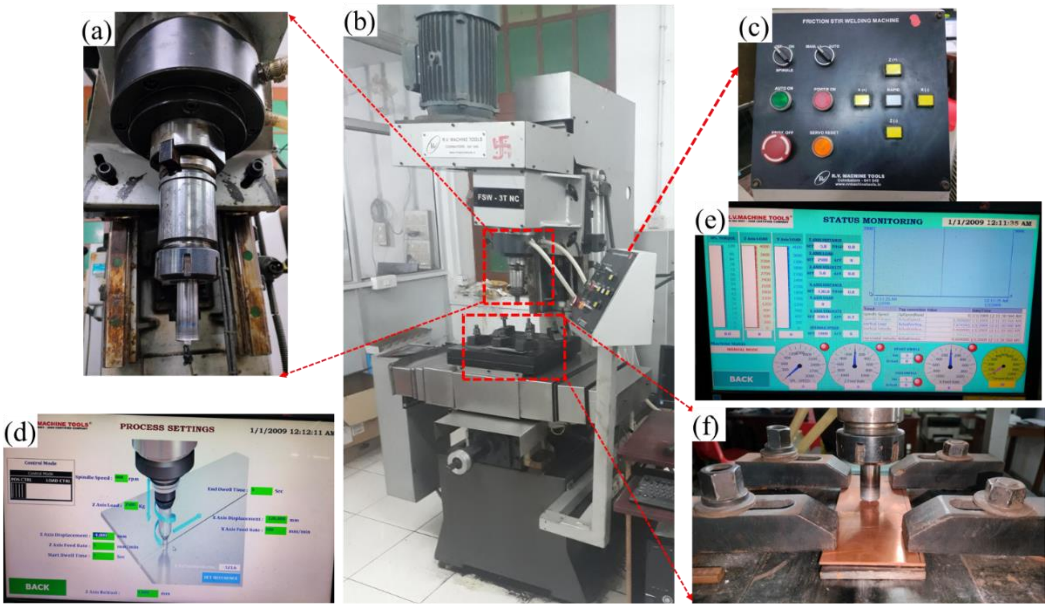

Figure 3-2 FSW machine setup

3.3 Selection of friction stir welding tool material

Once the selection of work material is done, a search for proper tool material for multipass cladding is initiated to meet the requisite conditions. An ideal tool material for performing multipass cladding should essentially possess sufficient elevated temperature strength and wear resistance so that the tool pin does not get worn out or deteriorate during multipass stirring action, or else clad regions with sufficient bonding cannot be reproduced. High speed steel tools or tools made up of tool steel are preferably suitable for low temperature softening materials like aluminium or magnesium, but have been reported to be unsuitable for carrying out welding or cladding of steel. Among the choices of materials available for welding or cladding on steel substrates are tungsten carbide (WC), polycrystalline cubic boron nitride (PCBN), polycrystalline diamond (PCD), and tungsten-rhenium alloys.

For FSW of steels, PCBN is a popular tool material due to its high strength, hardness, and stability at high temperatures. However, the PCBN tools also have the limitation of brittle fracture at higher traverse speed [197]. Tungsten carbide with cobalt binder is an excellent choice for joining steels due to its low cost and excellent toughness as compared to PCBN tools [198]. Tool material's characteristics depend upon the material to be welded, and some favourable tool material characteristics are:

- **Ambient and elevated temperature strength:** The FSW tool materials should be able to withstand the compressive load during welding. The tool should have sufficient shear and compressive strength at elevated temperatures to prevent tool fracture or deformation during welding.
- **Elevated temperature stability:** The temperature of the FSW tool while stirring during welding approaches the workpiece solidus temperature, and therefore, the tool must possess chemical and dimensional stability at elevated temperatures.
- **Wear resistance:** An FSW tool should have good tool wear resistance because excessive rubbing involved in FSW can cause tool wear, which can change the shape of the tool by removing tool features and thus rendering it unfit for subsequent welding.
- **Tool reactivity:** Tool material should not react with the environment or work material during welding and should be resistant to oxidation and changes in the surface properties of the tool. Surface reactions may lead to the formation of toxic substances like MoO_3. This resistance to the environment should also be exhibited by the tool material at elevated temperatures as well.
- **Fracture toughness:** The fracture toughness plays a significant role during tool plunge and tool dwell. When the tool first touches the workpiece, the generated local stresses are high enough to break the tool. Severe damage

to the tool is generally produced during tool plunge and dwell time. Therefore, care should be taken, and only materials with high fracture toughness should be used for making tools.

- **Coefficient of thermal expansion:** In the case of multimaterial tool pins and shoulders, large differences in the coefficient of thermal expansion between the tool pin and shoulder may lead to different expansions of one component in comparison to the other, resulting in stresses between the two and promoting tool failure.
- **Thermal conductivity**: Similarly, the FSW tool also needs to have less thermal conductivity to prevent heat loss and the transfer of heat to the main spindle bearing. The tool should also have a high frictional coefficient to promote the generation of heat.
- **Machinability:** FSW tools are often required to be given intricate shapes to the tool pin and shoulder, and hence, machinability plays an important role in getting the desired profile. A material that cannot be processed or given shape cannot be used for making tools.
- **Uniformity in microstructure and density:** Any local variation in microstructure and density within the tool material may produce weak regions within the tool and may lead to its failure.

3.3.1 Selection of tool material, tool shoulder and pin profile

In this work, two different tungsten carbide tools with different cobalt percentages, as shown in Table 2 have been chosen to perform cladding trial runs. The amount of binder, binder type, grain size of WC particles, and addition of other carbides largely influence the service life of the tungsten carbide tool. Generally, cobalt is used to bind

WC particles; however, recently, low-cost nickel has been used as a substitute for cobalt. Ideally, a tool with a low cobalt percent and a fine grain size of 1-2 microns proves to be suitable to carry out the cladding operation on a steel substrate. A higher percentage of cobalt or similar materials induces wear and deformation in the WC tool. The first tool, designated as ‘Tool A', is comprised of total (Co + Ni) > 10%. The ‘Tool A’ through which initial attempts were made failed miserably during cladding, as during the long run, WC particles dislodged from the pin face area, and it became unsuitable to continue the cladding. More analysis regarding tool degradation has been presented in chapter 4 Section 4.3. Midway tool degradation deteriorates the entire cladding as effective bonding gets affected between the mating surfaces. Hence, subsequent attempts were made with the tool designated as ‘Tool B’ having no nickel, and the cobalt percentage was also low (with a total percent of 4.95%) and was used to perform the cladding. ‘Tool B’ performed well during trials for long runs and did not degrade or weather, i.e., grains did not fall off from the pin-face region. Henceforth, tool B was found suitable, and therefore, the entire cladding was performed with tool B.

Table 2: Elemental composition of tungsten carbide tool A and tool B

Tool	Co% (Wt.%)	Ni% (Wt.%)	Fe% (Wt.%)	Hf% (Wt.%)	Cr% (Wt.%)	W% (Wt.%)
(Tool A)	3.29	6.81	0.33	1.95	0.17	87.4
(Tool B)	4.95	--	0.31	--	0.27	94.47

The tool geometry was selected such that it withstood the forces generated and the tool pin did not shear away during tool traverse. A proper pin length was also selected to ensure that the pin pierces through the clad metal and makes sufficient contact with the substrate material, ensuring a stirred, intermixed region. A tungsten carbide rod was ground on a tool grinder to produce the tool. Different tool profiles were tried to find the best profile that could ensure proper material stirring, distribution, and filling without any appearance of flaws. The tool with a concave tool shoulder and tapered pin profile, as shown in Figure 3-3a was initially tried, as this profile has been extensively used for welding [199]. The pin diameter at the pin face was D1, and the pin diameter near the shoulder was D2. Although the pin with a tapered profile ensured easy plunging of the tool, the volume of material stirred at the pin shoulder was different than at the pin face due to the difference in pin diameter. This tool profile resulted in a groove formation (as shown in Figure 3-3c), which might be attributed to the fact that the pin diameter was varying along the pin length. Therefore, for the rest of the experiments, a tool with a cylindrical pin and a straight shoulder was used. The final dimensions of the tool prepared were a straight shoulder with a diameter of 20 mm and a cylindrical pin profile with a pin diameter of 6.5 mm and a pin length of 4 mm, as shown in Figure 3-3b.

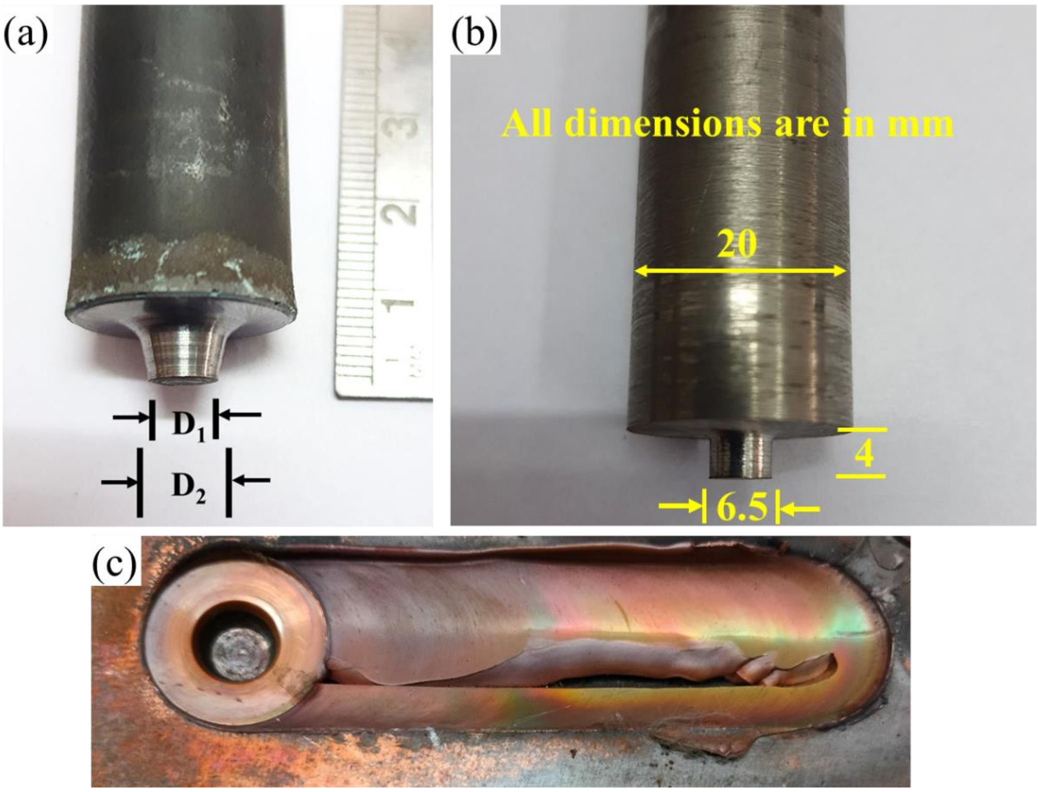

Figure 3-3 Illustration of (a) tapered tool pin profile depicting different pin diameter D1 and D2 (b) used tool profile (c) defect produced in the clad

3.3.2 Tool grinding setup

A tool grinding machine was used to prepare the tool profile effectively. A diamond grinding wheel was attached to the machine assembly to carry out the grinding operation. The grinding wheel had dimensions of 150 mm outer diameter, 31 mm bore diameter, and 6 mm width. It was comprised of an aluminium blank with diamond particles bonded to the outer periphery, as shown in Figure 3-4. The WC rod was held in the tool holder with a tapered shank collet chuck. The tool holder in the tool grinding machine was tightly gripped to prevent it from slipping. ER-40 collets with diameters corresponding to the tool blank used were used to grip the WC rod being ground. In this work, most of the tools were of 20 mm diameter; therefore, a 19–20 mm collet was used in the collet chuck. The tool holder was attached to the movable column of the machine and rotated at a low RPM of 1300, while the diamond grinding wheel rotated at a very high RPM of 3000.

To create a tool profile, the feed was given, and the tool holder was brought close to the grinding wheel to make contact. Material removal took place from the periphery of the WC tool upon rubbing, and the desired shoulder profile and tool profile were created.

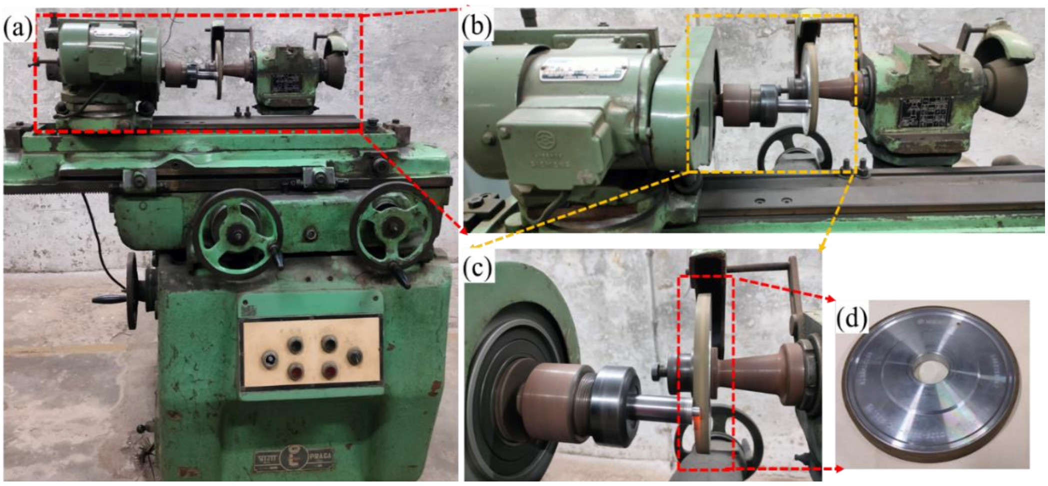

Figure 3-4 Tool grinding setup

A straight shoulder was obtained when the WC tool holder assembly was perpendicular to the grinding wheel, while a convex or concave-profile shoulder was obtained by tapering the tool holder assembly with respect to the wheel. A polygonal pin profile could also be generated by stopping the rotation of the tool holder and providing motion to the grinding wheel only, resulting in material removal from the face, and thereafter changing the face side by an angle accordingly to create the required number of faces in the polygonal tool.

With the aid of a diamond tool grinder, various tool geometries (convex, concave, straight) and similarly different pin profiles (cylindrical, taper) were also used in trial experiments to decide the optimum tool profile that could be used for experimentation.

3.4 Cladding trial runs

The available FSW setup has been primarily used for welding, mostly in butt and lap positions, and so the need arises to check the compatibility of machine and tool material for multi-pass cladding. Before attempting to clad copper on steel, initial trials were performed to clad copper on aluminium. Aluminium, being a low-melting, soft material, is easy to work with during the friction stir cladding trials. As aluminium is a low-strength material, less force is required during its friction stir cladding, reducing the chances of damage to the tool pin. Once the initial satisfactory results were obtained, trials were performed for cladding copper on steel.

3.4.1 Initial attempts for copper clad aluminium

To lightly load the machine, initial experiments were performed for cladding copper on an aluminium substrate. The copper was placed on top of the aluminium substrate, and both were securely clamped on the machine table. Several process parameters, like tool spindle RPM, tool speed, and tilt angle, were varied, and the cladded plates obtained were tested for their integrity or defect formation. However, the cladding of copper on aluminium posed several other challenges due to the low melting point of aluminium and the differences in properties of both materials, which made the selection of process parameters difficult. During the initial trail runs, at a high welding speed of 400 mm/min and a spindle speed of 1000 RPM, aluminium, which has a significantly lower melting point, melted down near the interface region and appeared as a re-solidified ball-like structure, as shown in Figure 3-5a. When trial runs were carried out with a longer pin length, the substrate material stirred too much and travelled to the top of the clad layer, as shown in Figure 3-5b. The presence of patches of substrate material at the top was observed due to the travelling of the aluminium substrate to top, which deteriorated the clad layer.

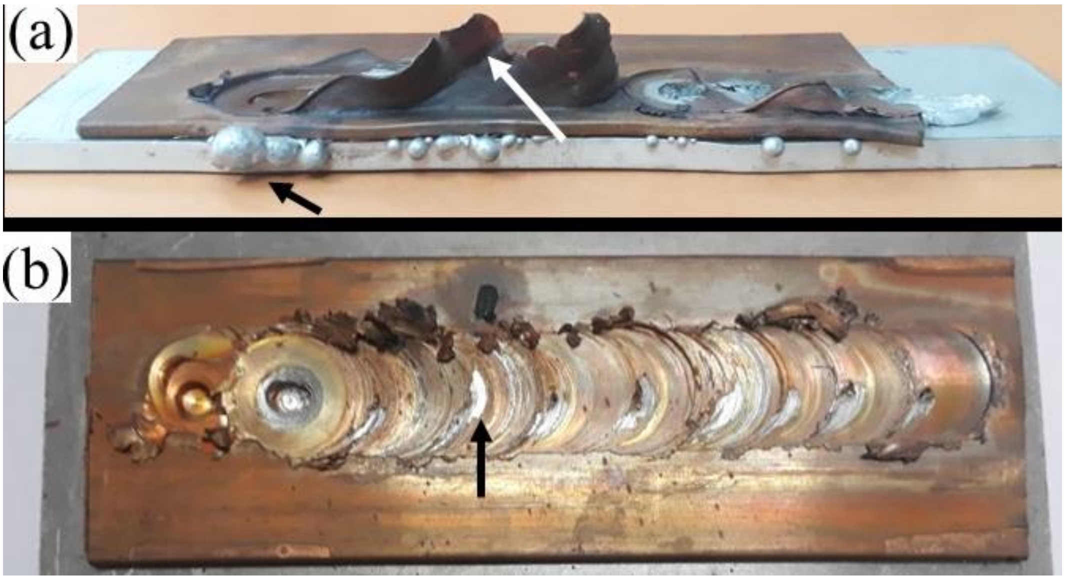

__Figure 3-5 Defects arising during cladding of copper on aluminium__

After several attempts to avoid excess heat generation, a medium spindle speed of 650 RPM and a high tool travel speed of 250 mm/min with a tool pin length of 2.8 mm were found to give satisfactory results. One such successfully prepared cladded sample has been shown in Figure 3-6.

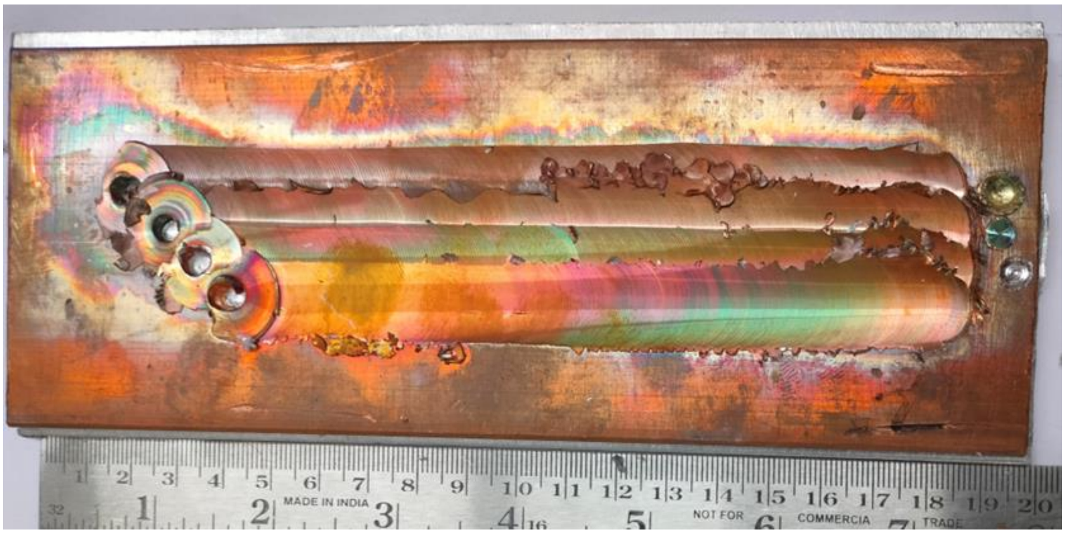

Figure 3-6 Successfully prepared copper clad aluminium plate

After successfully carrying out the cladding passes as per the opted process parameters, the cladded plate was removed from the machine fixture for visual examination. The inspection of cladded samples revealed no surface defects, and smooth ripples were observed on the top surface. There was no visible distortion of the clad plate or the underlying substrate. Aluminium was clearly visible inside the pin hole left at the end, indicating sufficient pin penetration despite the pin being shorter than the thickness of the copper sheet. Samples were taken out and prepared for microstructural examinations as per the standard methods. The microstructure of copper-cladded aluminium has been carried out, and results have been described in section 4.1 in the next chapter.

3.4.2 Trial runs for performing copper cladding on steel

After the successful demonstration of cladding copper on aluminium, efforts were made to work on the main objective of cladding copper on a steel substrate. To evaluate the feasibility of cladding copper on steel through FSW, copper was placed on top of the steel substrate and clamped tightly so that the plates would not slip under severe loads. Several trial experiments were carried out at different process parameters, i.e., varying the rotational speed, travel speed, and tilt angle in a wide range during the trial experiments. It was observed that the tool geometry and other process parameters, such as spindle speed, welding speed, and tilt angle, directly influence material flow and heat generation. Table 3 represents the experimental parameters at which cladding could be successfully obtained without any damage to the tool, and for the rest of the experiments, the parameters were fixed to the values represented in Table 3. A moderate spindle speed of 700 RPM and a low welding speed of 100 mm/min were found suitable to carry out the cladding operation. Higher spindle RPM and lower welding speed than this parameter resulted in excessive heat generation, consequently making the clad material too soft and causing it to flow in a lateral direction. Contrary to this, too low spindle RPM and a higher welding speed than this parameter result in insufficient heat generation for the thermo-mechanical processing necessary for cladding. Similarly, a tool tilt angle of 1.5 degrees worked well for the cladding requirements, which is another vital parameter that helps to ensure proper stirring.

Table 3 Process parameters used for FSW cladding of copper on steel

Tool	**Spindle speed**	**Travel speed**	**Tool tilt**	**Shoulder Diameter**	**Pin diameter**	**Pin length**
WC	700 (RPM)	100 mm/min	1.5°	20 mm	6.5 mm	4 mm

3.4.3 Plunge depth optimisation during copper clad steel

The optimal plunge depth needs to be evaluated before performing the cladding operation of copper on steel. This differs from the case of copper cladding in aluminium, as steel offers more resistance to tool pin plunging and, hence, experiences a larger magnitude of forces in comparison to aluminium. The large impact of forces may also break the tool pin, which was not generally observed in the case of aluminium.

For each new tool pin, it necessitated an evaluation of the optimum tool plunge to reach the necessary depth, as a very high tool plunge can lead to excessive flash and can damage the tool pin. The steps followed while optimising the necessary tool plunge have been demonstrated in Figure 3-7. With the new tool pin, a low tool plunge was initially selected intentionally to avoid accidental damage to the freshly prepared pin at a larger plunge; as a result, a very narrow plunge depth can be seen in step 1. Subsequently, the tool plunge was gradually increased, resulting in a gradual increase in plunge area during steps 2–5. During these steps, the exit hole consisted of all copper, indicating no contact of the tool pin surface with the steel substrate. Based on the previous outcomes, the tool plunge was further increased for step 6, which resulted in the appearance of a flash around the periphery. It is also worth noting that the steel substrate can be clearly seen in the exit hole, indicating that the tool pin has pierced through the copper layer and into the steel substrate. The flash that appeared was unacceptable; therefore, further trail runs were carried out to ensure complete penetration of the cooper layer without generating excessive flash. Consequently, a lower tool plunge in comparison to step 6 was tried in step 7. Although lowering the tool plunge removed the flash, it resulted in an incomplete circle width in the exit hole, as shown by the arrow in step 7. In the next step, i.e., step 8, the plunge was increased, resulting in a sound-clad appearance without any defects. This plunge condition was again tested in the next step to verify the repeatability of the selected plunge. The plunge depth for step 9 was similar to step 8, i.e., without any process defects. Thus, this plunge was finalised to carry out the multiple passes for copper-clad steel.

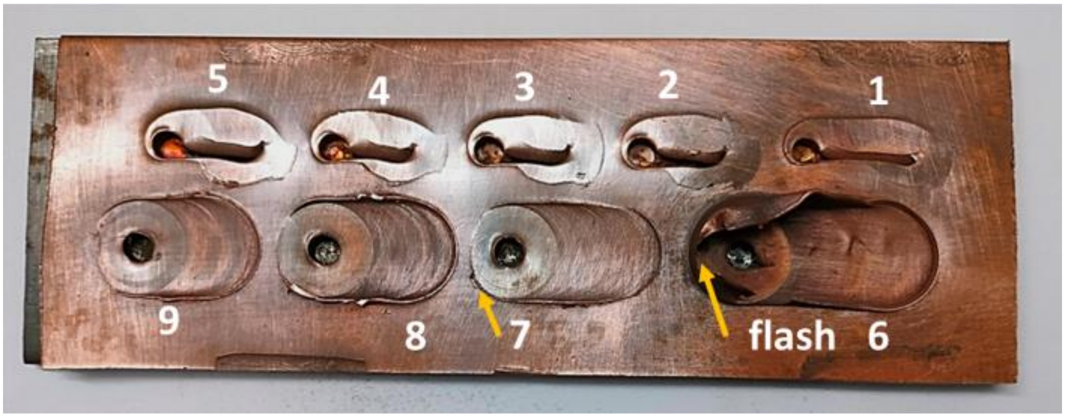

Figure 3-7 Steps for plunge depth optimisation during copper clad steel

3.4.4 Tool offset distance selection

As with every other process, there are several parameters that need to be taken care of for a successful clad using FSW. Apart from the process parameters like spindle speed (RPM), tool travel speed (mm/min), and tilt angle (°), it is of prime importance to decide the tool offset that needs to be provided for joining the material in the region that has been cladded. In this work, the effect of tool pin offset variation during friction stir cladding has been examined. Smaller tool offset distances require a greater number of passes to ensure covering the entire width of the plate, thus consuming a larger amount of time, along with overutilization of machines, manpower and resources, leading to poor productivity. On the contrary, a very large tool pin offset hampers the joining efficiency by leaving the area between two passes unbonded and providing only superficial bonding between copper and steel. An optimum offset value can lead to higher productivity by carrying out only the essential number of passes to clad the substrate and avoiding unnecessary overutilization of resources. Thus, offset distance selection proves to be a key aspect for cladding through FSW. Hence, the effect of tool pin offset on multipass cladding has been dealt with here. The suitable offset distance was selected by preparing clad plates with different offset distances between the passes. The tool pin offset distance was varied from 6 mm to 12 mm. Cladded plates were produced by keeping the tool offset distances at 6, 8, 10, and 12 mm, and the plates thus produced were evaluated for proper bonding.

3.5 Characterization of cladded samples

Numerous different characterization techniques were used to evaluate the soundness of the clad produced and to thoroughly test its quality. Visual inspection, microstructural examination, radiography, and mechanical tests like tensile testing, microhardness evaluation and guided bend tests were carried out. The details of these characterizations have been described in this section.

3.5.1 Visual inspection

Visual inspection serves as one of the preliminary screenings for the prepared samples. Many kinds of flaws, which are on the outer surface of the prepared clad, are visible to the naked eye, get detected and are discarded. This is generally a fast process, and its success depends on the expertise and experience of the invigilator. Many flaws that do not appear on the outer surface can qualify for this invigilation and need to be further investigated properly.

3.5.2 Temperature measurement

Temperature measurement gives a more in-depth evaluation of the cladding operation. In this work, temperature measurement was recorded through a non-contact FLIR (forward-looking infrared) camera (FLIR-E75, USA) to evaluate the heat generation during cladding operation. The measurements were taken during the plunge, during tool travel and after the tool withdrawal stages at an interval of 10 seconds.

3.5.3 Radiography of cladded samples

In the welding industry, one of the most popular non-destructive testing methods to assess the integrity of weld joints is X-ray radiography. This testing technique uses X-rays to produce an image of any interior weld defects that may be present. X-rays have a very high penetrating capability due to their very small wavelength. When X-rays are aimed at the weld plate, some rays get absorbed by the material, while the remaining X-rays pass through it and are detected by the detector (photographic film) that is positioned behind the plate. The density and structure of the material influence how much X-ray energy gets absorbed. The quantity of absorbed energy at a particular spot varies at locations where the welded plates have any discontinuity or dissimilar material trapped inside. The photographic film depicts the changes in the absorbed X-rays in different shades of black and white. Thus, X-ray photographic film helps to detect any kind of discontinuity that is present within the weld.

In this work, X-ray radiography of the produced cladded samples was carried out following the ASTM E1032 standard using a Gamax Inc. industrial X-ray unit (Model 333) (focal spot: 1.5 mm). The X-ray images were acquired after an exposure time of 1.8 seconds. During X-ray radiography, the cladded samples were irradiated from the top and analysed for any kind of flaw.

3.5.4 Metallography of cladded samples

Metallography serves as one of the important aspects of this characterization section, as it enables one to observe the metallurgical changes that evolved in the transverse or any cross section of the cladded samples. Samples were prepared for optical microscopy and scanning electron microscopy. Proper sample preparation is very important to obtain good microstructural and macrostructural images and to evaluate the bond quality between the clad layer and the substrate.

3.5.4.1 Sample preparation for microscopy

After careful visual examination of the cladded plates, a 10 mm-wide strip was extracted from the centre of the cladded region through a wire EDM machine. Wire EDM ensures less thermal and mechanical damage to the extracted samples. To assist in holding the specimens during polishing and etching, the specimens were hot moulded in bakelite by using a hot moulding machine. The transverse cross sections were on the exposed side, while all other regions of the samples were under bakelite. The transverse cross section of the cladded samples was polished with different grades of emery paper, ranging from grade P120 to grade P2000. Once switching from a coarser grade of emery paper to the next finer grade, the samples were rotated by 90 degrees, and the polishing direction was kept perpendicular to the previous direction of polishing. After the polishing was complete on the finest grade of emery paper (i.e., P2000), cloth polishing was done with abrasive alumina paste (0.05 µm particle size) and water for steel, whereas brasso solution along with kerosene oil was used for polishing copper. The prepared transverse cross section appeared as shown in Figure 3-8. The microstructure evaluation was carried out on the transverse cross section of the cladded plates following the ASTM E407 standard. To expose the grain boundaries of mild steel, a 5% Nital solution (ethanol 100 ml, concentrated nitric acid 5 ml) was used, whereas for etching copper, an etchant consisting of an equal volume of distilled water and nitric acid was used. Attempts were also made to etch both copper and steel concurrently. A combination of 10 parts HCl and 1 part nitric acid worked well and etched both copper and steel simultaneously.

Figure 3-8 Prepared transverse cross section sample for metallography

3.5.4.2 Optical microscopy

To reveal the intensity of bonding between the clad material and the substrate material and to understand the development of microstructure after cladding, the metallographic study of the cladded samples was necessary. The microstructure of the cladded sample also helps in predicting the behaviour of weldment, understanding the effect of multipass friction stir cladding on the clad and substrate, and finding the grain size. This also helped in predicting, up to what depth the effect of multipass cladding was present in the substrate plate. The presence of any micro voids could also be traced with the help of a metallurgical examination. The images from the transverse section of the cladded samples were taken by an optical microscope (Dewinter, classic PL) at various magnifications ranging from 100X to 600X. A computer was attached to this microscope to capture the images.

3.5.4.3 Scanning Electron Microscopy

Compared to conventional microscopes, the scanning electron microscope offers many advantages. Owing to its wider depth of field, a larger area of the specimen can be in focus at a time, and due to its high resolution, closely spaced features can be magnified to very high levels [200]. Another advantage that SEM offers is to give micrographs of 3D objects, like fractured tensile surfaces or uneven surfaces, which is not possible through optical microscopy, which requires flat specimens for microscopy.

Modern SEM offers the facility of elemental mapping and elemental composition analysis, giving insights into the composition of the material. Most optical microscopes offer a magnification of up to 2000X only, while the magnification achievable through SEM can reach up to 100000X. The scanning electron microscope (ZEISS EVO 18 Research, Germany) equipped with the EDS accessory (voltage 20 kV, working distance 11.5 mm, signal SE) has been used for this work to evaluate the metallurgical bonding produced by the cladded sample. HRSEM facilities were carried out (Model: Nova Nano SEM, voltage 15 kV, working distance 4.9 mm, signal SE) and (Model: EVO SEM CARL ZEISS, voltage 20 kV, working distance 10 mm). Similarly, elemental mapping across the transverse cross section helped in understanding the cross-boundary metal transfer between the clad material and the substrate.

3.5.5 Microhardness testing

Hardness is the resistance of a material to localised plastic deformation. Microhardness testing is highly helpful for determining the hardness of thin sections, measuring individual hardness within a larger matrix, or determining the hardness gradients of a part along the cross section. Here, the microhardness tests were carried out to observe the variation in hardness behaviour of the clad copper and steel substrate post cladding across all regions (transverse cross section face, transverse cross section across the interface, and copper clad top). Vickers microhardness measures the hardness of material by measuring the size of the impression produced under load by a pyramid-shaped diamond indenter. The indenter used in the Vickers hardness test is a square-based pyramid whose opposing sides come together at the top at an angle of 136 degrees [201].

The test was performed by placing a flat specimen of the cladded sample exactly below the indenter. The Vickers microhardness test was carried out on the transverse section of the cladded specimens, as shown in Figure 3-9, as well as on the top surface of the cladded plates following ASTM E92 standards. A load of 25 grams was used for copper and 50 grams for mild steel to observe the microhardness variation across all layers of copper-clad and steel substrates.

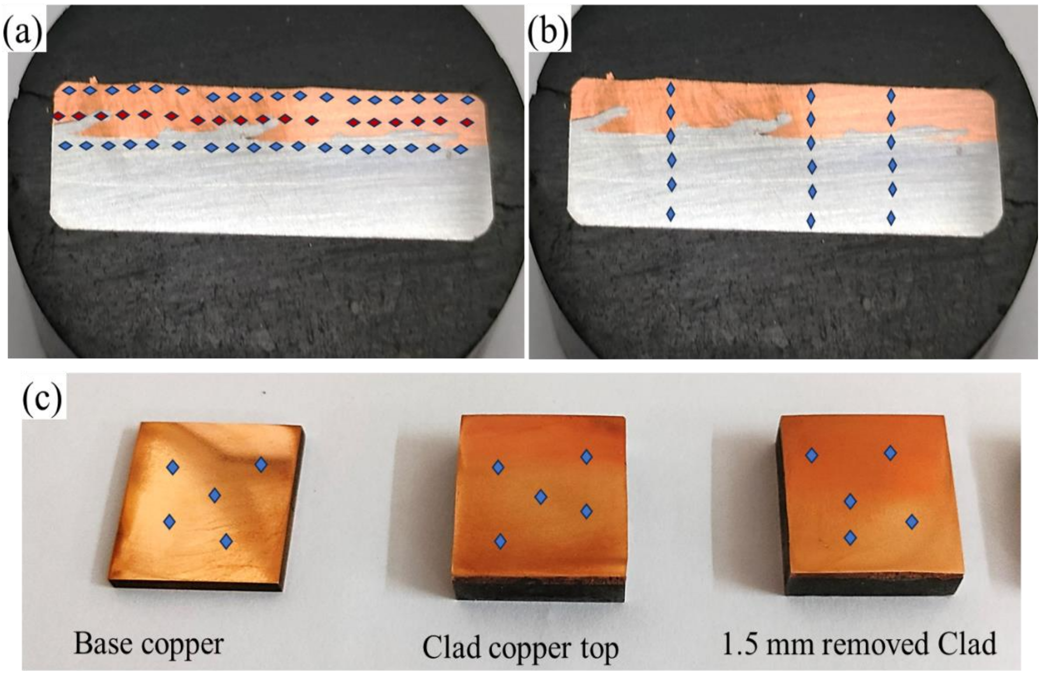

Figure 3-9 Microhardness evaluation at locations chosen on the sample

A microhardness test was also carried out along the cross section across the clad layer, with systematic indentations made on the copper layer, interface, and steel substrate. Indentations were made along a linear profile running from the substrate, through the interface until the copper-clad layer. An interval of 0.5 mm was kept between each indentation to ensure sufficient distance between each indentation. The microhardness testing across the profile was done at a load of 50 grams. Microhardness analysis was also carried out on the top surface of base copper and clad copper top to estimate the hardness of the cladded sample.

3.5.6 Tensile testing

The tensile test is one of the most preferred mechanical tests for finding the strength of a component, including a welded one. Tensile specimens are classified into two types: flat specimens and round specimens. Flat test specimens are generally made when the specimen is in sheet or plate form, while round specimens are made when material is available in bulk form. In this study, uniaxial tensile tests on longitudinal flat specimens were carried out as per ASTM E8 standards [202]. The tensile test was carried out to evaluate the tensile strength of the produced clad joint with respect to the base material. The test was carried out on an Instron 8801 servo hydraulic testing system at room temperature with a cross head speed of 1 mm/minute. Three sets of tensile test specimens were prepared for each condition, as shown in Figure 3-10 along with their dimensions. The thickness of the steel-copper cladded plates was approximately 9 mm, and a tensile specimen of 6 mm thickness was obtained from the cladded sample by milling the substrate plate from the opposite side. A tensile test was also performed to evaluate the strength of the copper used in cladding and the strength of the substrate material (steel). The thicknesses for copper specimens were 3 mm, and those of the substrate materials, i.e., steel, were 6 mm.

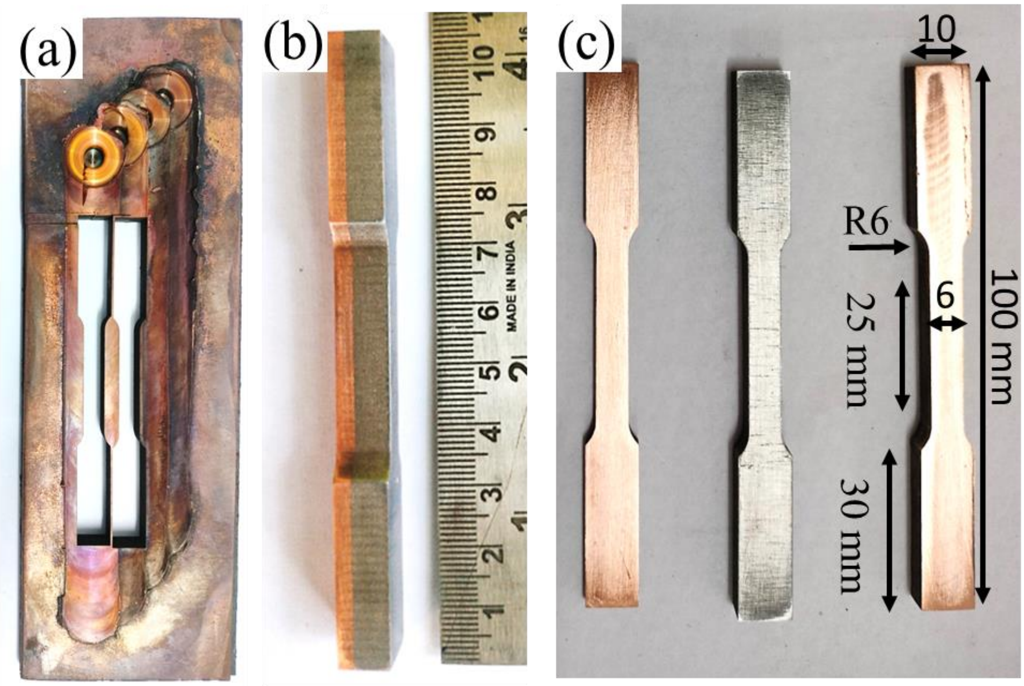

Figure 3-10 Illustration of (a) cladded plate demonstrating location of extraction of tensile specimens (b) side view of cladded tensile specimen (c) prepared tensile samples of copper, steel, and copper clad steel

3.5.7 Guided bend test

A guided-bend test is a destructive technique used to determine the quality of a weld joint at the face and root of a welded joint. During this test, a flat specimen is placed on two rollers located equally from the line of force application. The specimen is subjected to a centrally applied force through a circular-shaped plunger, which bends the specimen into a U-shaped die to bend the plate into a U shape. The bent specimen's convex surface is checked for cracks or other open flaws [203]. A guided bend test is carried out to investigate whether proper bonding has taken place between the clad and substrate or not. This is very essential to ensure that the clad does not fail or delaminate during service.

A guided bend test was carried out as per ASTM E190 [204], and three sets of samples were prepared for testing in each condition (face bend-substrate up), (root bend-substrate down), and (side bend-transverse section) from the mid-section of cladded plates using a wire EDM machine. The three views of samples prepared for bend tests have been shown in Figure 3-11a. Each longitudinal bend test sample was 150 mm x 10 mm x 9 mm in dimensions. The extracted cladded samples were placed on top of roller supports equidistant from both ends, as shown in Figure 3-11b, and bent in a U shape by applying force to the midsection with the help of a plunger. The copper-clad steel samples after bending were evaluated for the possibility of delamination or the occurrence of any cracks or defects in the cladded interface.

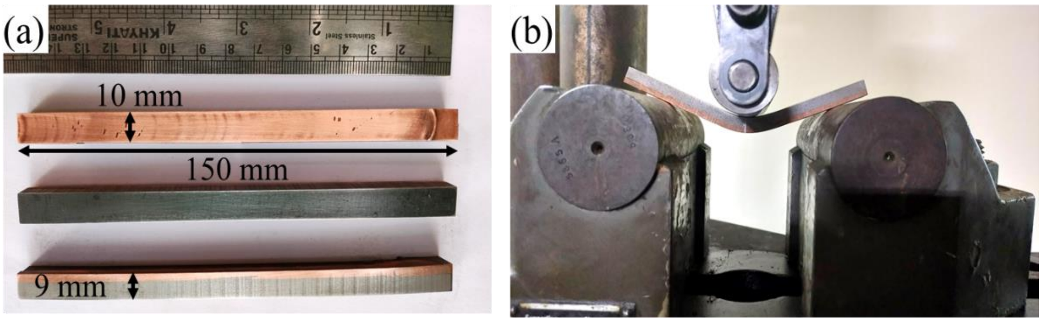

Figure 3-11 Prepared samples (a) for guided bend test (b) undergoing bend test

3.5.8 XRD analysis

X-ray diffraction (XRD) is a non-destructive technique that gives information about chemical composition, crystal structure, crystallite size, lattice strain, and preferred orientation. X-ray diffraction is the result of constructive interference between X-rays and a crystalline sample. A variety of materials, including powders, solids, thin films, and nanomaterials, have been analysed using XRD [205].

XRD analysis was carried out to determine the phases present on the top and on the transverse cross section of the copper cladded steel plate. During cladding, the possibility of contamination of the top clad surface with substrate material is high, which can deteriorate the clad effectiveness. Therefore, to rule out the possibility of such formations, along with the probable intermetallic formation at the interface, an XRD evaluation was performed. Specimens were taken out for XRD analysis from the midsections of cladded specimens. The size of the specimen was 10 mm x 10 mm for XRD on the top surface, while the combined width of the specimen was 9 mm x 15 mm for the specimens taken out for XRD analysis on the transverse section. XRD analysis was performed under a working current of 15 mA and a working voltage of 40 kV with a step width of 0.02 degrees and a scan range of 5–100 degrees with a scan speed of 7 deg/min.

3.5.9 Corrosion test

Proper evaluation of corrosion helps in determining the behaviour and life span of any structure, which can prevent any catastrophic failure [206]. In this work, cladding of copper has been done over a steel substrate through FSW, which protects the beneath mild steel substrate. Proper evaluation of corrosion behaviour helps in determining the resistance of the material to the environment and predicting its service life. Cladding can prevent any catastrophic failure of the mild steel substrate underneath from corroding. Therefore, to analyse the effectiveness of clad copper against corrosion with respect to base copper and base steel, this evaluation has been carried out.

Three different surfaces were chosen for corrosion analysis: base copper, clad copper top surface, and clad top copper surface after 1.5 mm removal. One square cross-section of 25 mm x 25 mm was extracted from base copper and two from copper-cladded steel. Out of the two samples extracted from copper-clad steel, one sample was milled, and 1.5 mm of the top copper clad layer was removed to expose the underlying material. All three samples were polished and tested for their corrosion behaviour. The freshly polished samples and samples after the corrosion testing have been shown in Figure 3-12. Figure 3-12a is for base copper, Figure 3-12b is of clad top copper, and Figure 3-12c comprises the clad top after 1.5 mm removal. A potentiodynamic test was performed on an Autolab 302N machine with three-electrode arrangements. Base copper and the cladded copper samples being tested were used as working electrodes. Platinum mesh and saturated calomel electrodes (SCE) were used as counter and reference electrodes, respectively. A 3.5 percent NaCl electrolyte was used to carry out the tests at room temperature. The working electrode was allowed to attain steady-state potential in the test solution before carrying out electrochemical studies. With respect to E_{ocp}, potentiodynamic scan measurements were taken at a scan rate of 2.45 mV/s from a cathodic potential of -1.27 V to an anodic potential of +1 V. E_{corr} and I_{corr} values were obtained from the plotted tafel plots.

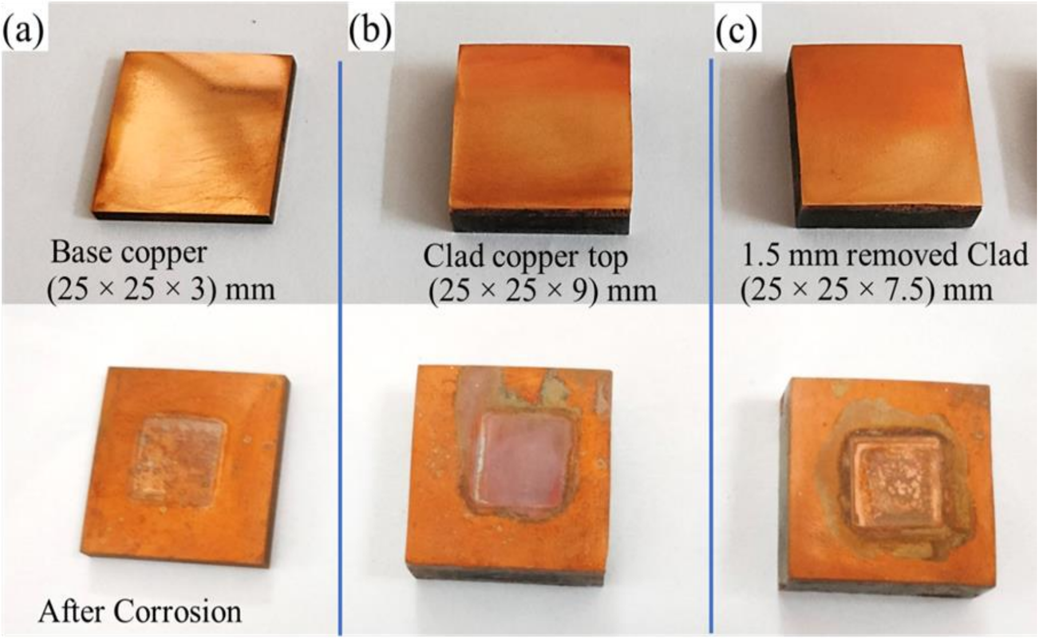

Figure 3-12 Corrosion samples pre and post corrosion (a) received copper (b) clad copper top (c) clad copper top after 1.5 mm removal

3.5.10 Atomic force microscopy

AFM (atomic force microscope) is a non-destructive technique used for the surface topography of materials, and it proves to be an important tool for corrosion. The AFM offers a three-dimensional surface profile as opposed to the scanning electron microscope's two-dimensional projection or image of a material. AFM produces a 3D image of the sample surface with quantification of surface roughness (i.e., average roughness, root mean square roughness, maximum peak to valley height), giving a pictorial representation of the protective film at nanoscale [207]. The surface roughness parameters affect the performance of the clad layer. The progression and severity of corrosion damage, including those due to biological factors in the marine environment, depends on the surface topography and roughness, which can be accurately measured and analysed by AFM. AFM consists of a cantilever with a sharp tip, and as the tip scans over the surface, the interactions between the AFM tip and the features on the surface cause displacement of the cantilever. The AFM creates topographic images of the surface by plotting the laser beam deflection as its tip scans over the surface [208]. AFM operates in three modes: contact mode, noncontact mode, and tapping mode. In contact mode, the probe is in continuous contact with the sample while the probe raster scans the surface. In non-contact mode, the cantilever oscillates near the surface of the sample but does not contact it. The oscillation is slightly above the resonant frequency. While in tapping mode, the cantilever oscillates at or slightly below its resonant frequency [209].

AFM (atomic force microscope) 2D and 3D pictures were taken in order to compare the topographical features of the base copper with respect to the corroded copper surfaces of the base and cladded ones. AFM analysis was carried out on (maker: NT-MDT, model: NTEGRA PRIMA). After performing the potentiodynamic polarisation test, SEM and AFM pictures were evaluated in order to compare the morphological features of the base copper with respect to the corroded copper surfaces of the base and cladded samples.