



## Combination of SX Steel with Copper Intermediate and GR5 (Ti6Al4V) Titanium Alloy by Diffusion Welding

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### ABSTRACT

Diffusion welding, also known as solid state welding or pressure welding, is a type of welding process that involves bonding two metal surfaces together by applying heat and pressure, without or using any interlayer material. In this study, the effect of GR and SX materials of welding temperature on the connection is examined with the diffusion welding method using a copper intermediary layer. The material pairs are combined with a pressure of 5 MPa and a fixed welding time of 60 minutes in an argon gas environment in a silicon bar oven at the temperature of 980, 1000, and 1020°C. The SEM-EDS-AFM analysis of the combined samples is performed, the metallographic structure of the intermediary layer in the diffusion-affected region is examined by the optical microscope, and the possible phases are tried to be determined by the XRD analysis. The micro stiffness values of the connection area are analyzed.

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## Introduction

Titanium materials have a major role in industrial aviation in the commercial market because of their resistance to high strength and temperature corrosion [1] - [3]. GR5 (Ti6Al4V) materials are  $\alpha+\beta$  Ti alloys, and these alloys contain between 10-50% of the  $\beta$ -phase at room temperature [4]. The SX 316 is a version of the SX 304 with enhanced molybdenum and a slightly higher nickel.

The resulting composition of this SX 316 gives the steel much higher corrosion resistance in many aggressive environments. Molybdenum makes the steel more resistant to cavitation and cracks corrosion in chloride-contaminated environments. A lower overall corrosion ratio in slightly abrasive environments provides the steel with a good atmospheric corrosion resistance in dirty marine atmospheres [5].

The most important alloy element to help ensure corrosion resistance in stainless steel is chromium, and it must be weighted at least 11% of the composition. In addition of nickel and molybdenum to the chemical compound, the corrosion resistance of steel is increased further [6].

The diffusion source is a process where welded parts are in close contact by heating up to a certain temperature under controlled pressure. The diffusion bond, as its name suggests, contains the internal diffusion of atoms along the interface of the solid and sometimes liquid welding, and is a local plastic deformation and maximum surface approach that allows the creation of atomic diffusion and high-strength connection between two welded parts [7] - [9].

The production of quality diffusion weldings with low plastic deformation appears to be possible by using a soft intermediate layer between inter surfaces and GR2 and GR5-rich titanium alloy materials, using the aluminum arc, constant pressure in the argon protective gas atmosphere (5 MPa), constant time (55 min) and 850, 900 and 950°C are combined with diffusion welding method in a problem-free manner [10], [11].

Titanium and alloys exhibit different characteristics if they are in the martensite alpha structure and they have a needle shape. For example, alloys with a coaxial grain structure have high sponges and strength, high styling capability,

and high resistance to tensile corrosion cracking, while alloys with needle grain construction have excellent creeping resistance and good fracture toughness [12].

Aydin, Hidiröglu, Kaya, and Kahraman have determined that the diffusion of titanium to copper and copper to titanium happens at different distances and densities. They have observed that it is caused by atomic diameters, activation energies, and cage structures [13].

Akca and Gürsel have investigated the impact and importance of the intermediate layer in diffusion welding. They have found that one of the best intermediate layer materials is copper, based on their results using different metal duos and different metal intermediate layers because copper has high thermal conductivity and the ability to transmit which allows higher heat flows [14]. Akca and Gürsel have combined aluminum with titanium alloy by using diffusion welding at a constant temperature of 560-600-640°C for 60 min and under the influence of argon gas. They have determined that hardness values increase when the welding temperature increases and titanium begins to take place in the  $\beta$ -phase structure [15].

AISI 304 stainless steel has been combined successfully with (86.86 SiO<sub>2</sub>%) porous ceramic material with high silicon by diffusion welding using an intermediate layer, but there has not been enough fusion at temperatures below 850°C [16]. Atasoy has argued that the optimal intermediate material parameters should be selected to combine titanium and low-carbon steel materials with diffusion welding and have obtained the highest intermediary strength in samples combined with 90 minutes and at 850°C in the study [17].

Metal corrosion caused by acids creates a regional problem near the source of contamination. The main cause is due to dry air build-up. For metal surfaces in contact with the soil, the increase in acid in the soil is proven by data to be a factor that increases the risk of corrosion [18].

The Vickers test is usually easier to use than other hardness tests because the necessary calculations are independent of the indent size and the indentation can be used for all materials regardless of hardness. Precision cutting is used to separate samples or cut a sample into a very precise position [19], [20]. Surface roughness is one of the reasons that affect the quality of the diffusion welding. Roughness affects the time required for maximum bonding between welded surfaces. During the fusion process, especially the wave-length long rough is important. High pressure and long welding time must be applied to eliminate gaps. Because diffusion depends on temperature and time [20].

In diffusion welding studies using a cart, the regions with the highest hardness values are those close to the interlayer [22]-[26].

Increasing the temperature in interlayer diffusion welding works is positive for the formation of more intermetallic phases [27].

## Material and Method

### Materials

GR5 (Ti6Al4V) is a material commonly used in aerospace and space, biomedical and automobile sectors, consisting of pure titanium at a minimum of 99% purity. Besides being lightweight, GR5 is separate from similar materials with its ability to resist corrosion at high levels and show high strength. SX steel is high silicon-content austenitic stainless steel with the ability of good quality of welding and excellent corrosion resistance in high concentration sulfuric acid and nitric acid. SX is used in areas such as acid coolers, acid piping systems, acid distributors, acid towers and tanks, pumps, nozzles, internals, strainers and mesh pads. With improving technology, the need for steel-titanium materials that are resisted to acid and made with welded joint is increasing. Commercially supplied for distribution welding, GR5 and SX steel are prepared by cutting underwater on the precision cutting machine of 10x6 mm. At 99.5% purity of 30 $\mu$ m thickness, the hand-held Cu foil in Alpha Aesar is used as searchable. The chemical components of the materials are given in Table 1. The cutting stages of the samples are shown in Figure 1.

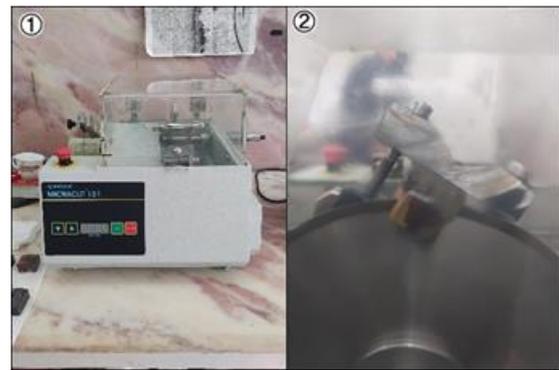


Figure 1. 1) Precision cutting machine, 2) Cutting metals.

Table 1. Chemical components of SX and GR5 materials

Element	Fe	C	Si	Mn	P	S	Cr	Ni	Mo	Cu
SX	55	≤0.025	5.0	0.5	≤0.045	≤0.030	17.5	19.5	0.4	2.0
Element	Ti	V	Al							
GR5	93.32	3.5-4.20	2.68							

Table 2. Mechanical properties of materials.

	SX	GR5	Cu
Density (g/cm <sup>3</sup> )	7,9	4,51	8,96
Melting Point (°C)	1540	1660	1085
Modulus of Elasticity (MPa)	200	110	120
Thermal Conductivity(W/m°C)	11	21,9	413

**AFM Analysis**

The surfaces of the materials to be combined with the diffusion welding are polished with 180-240-360-400-600-800 and 1000 mesh water based sanding and then the surfaces are made to be ready for surface topography application with the AFM (Atomic Force Microscope) device. Surface analysis and surface roughness measurement with AFM have been applied to all three material surfaces. The wavelength difference of roughness on the surface of all three materials has been noticeably

observed with AFM analysis. When the wavelength of the SX and copper is examined, a homogeneous surface image is obtained. However, when looking at the wavelength of titanium surface roughness, it has been observed that a more heterogeneous surface formed than the other two materials (Figure 2, 3, 4). The reason is thought to result from the fact that surface roughness, arithmetic average deviation (RA), quadratic average roughness, and the value of Rq (RMS) on GR5 are higher than the other two material types (Table 3).

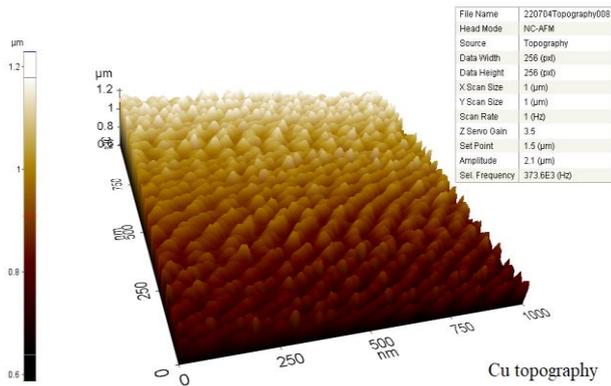


Figure 2. AFM images of GR5 materials topography.

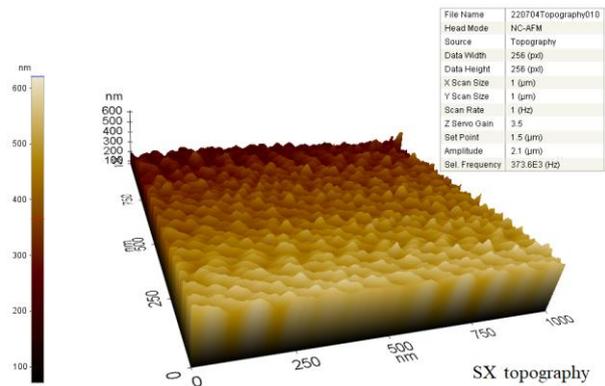


Figure 3. AFM images of SX materials topography.

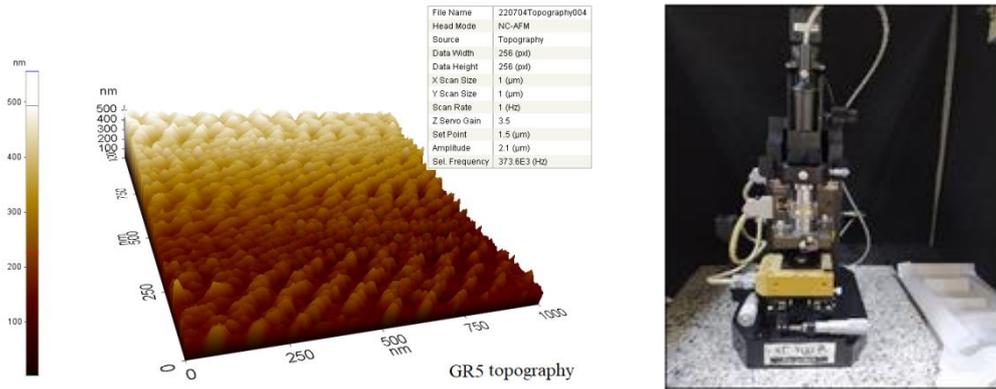


Figure 4. AFM images of GR5 materials and Atomic Force Microscope.

Table 3. Surface roughness values (µm).

Materials	R <sub>a</sub>	R <sub>q</sub>
Cu	0.0039	0.0046
SX	26.854	33.782
GR5	53.168	60.940

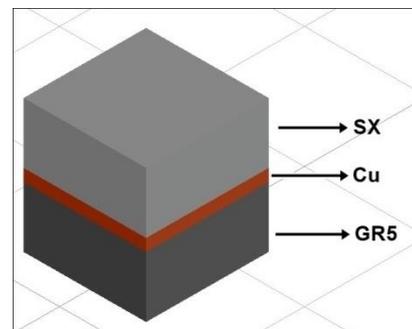


Figure 6. Schematic view of samples prepared for diffusion welding.

### Diffusion Welding

Three groups (M1, M2, and M3) for experiments and three samples for each group are prepared and the average of the applied test results is calculated. The samples are welded with specially prepared equipment for diffusion welding, sending argon gas with a purity of 99.9% to the high-temperature oven at a 3 lt/min flow rate (Figure 5).



Figure 5. Diffusion welding furnace.

Test samples are welded at temperatures 980, 1000, and 1020°C in 60 minutes at 5MPa constant pressures. The welding parameters applied to the samples are given in Table 4.

Table 4. Diffusion Welding Parameters

Parameters	M 1	M 2	M 3
Pressure(MPa)	5	5	5
Temperature(°C)	980	1000	1020
Time (min.)	60	60	60

### Microstructure Analysis

Samples combined with diffusion welding are cut perpendicular to the intermediate surface with a precision cutting device with a diamond disc for microhardness measurement (Figure 6). The surfaces are then sanded with 400- 600-800-1000-1200 and 2000 grit respectively, then polished with 9, 6, and 3µm diamond paste, and dried with dry air (ultrasonic) by applying the alcohol and high-

frequency sound waves to a liquid-filled tank. Samples prepared for analysis with the optical microscope (Nikon Epiphot 200 Inverted) are etched in solution 6ml HF, 9ml HNO<sub>3</sub>, and 85ml H<sub>2</sub>O (King water) and dried with hot dry air after they are washed with distilled water and cleaned with alcohol.

## Microhardness Measurements

The microhardness values of the samples are measured in the AOB Vickers hardness device (Figure 7). Hardness measurement is carried out by applying the 50 grams load for 10 seconds. Average microhardness values are taken after approximately 9 different points and 3 measurements from each point at approximately 10 $\mu$ m intervals are carried out (Figure 8).



Figure 7. Vickers microhardness measuring machine [21].

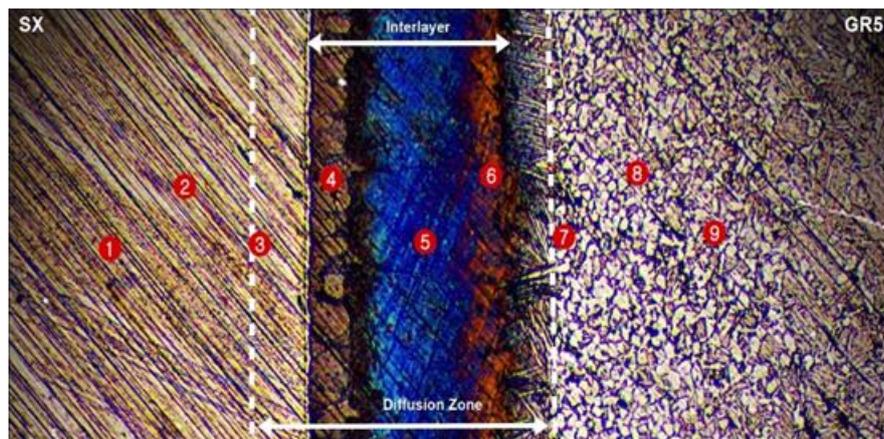


Figure 8. Microhardness measurement points.

## Results and Discussion

### Microstructure Results

The samples are successfully combined with the diffusion welding method using Cu intermediate layer. When the microstructure images of the test samples are examined, dense diffusion zones are observed in M3 samples at 5 MPa pressure, 1020°C, and 60 minutes of standby time. The intermediate layer does not create contrast, the microstructures on both sides of the intermediate layer are the opposite of each other. In the sample shown in Figure 4, Cu intermediate layer, the area affected by diffusion, and the martensite-like needle structure directed from GR5 materials to the intermediate layer are observed. In the welded samples, a good intersurface connection with optimum mechanical properties is obtained (Figure 9). In

addition, no gaps have been observed in the area adjacent to the main materials on both sides of the copper interface and no cracks have occurred in the joint area. During diffusion bonding, titanium, copper, iron, chrome, and nickel atoms have mixed in the copper interface. At welding temperature, iron and nickel have formed continuous solid solutions in irregular shapes, and the elements Ti, Ni, Cu, Fe, and Cr have produced the possible phase distributions seen in Table 3 along the intermediate layer. It has been observed that more titanium atoms are enriched in the middle and depleted in the area adjacent to the main metal. The distribution of iron has shown a model similar to titanium. The Cu component is concentrated in the center section and less Cu is detected on both sides of the center section.

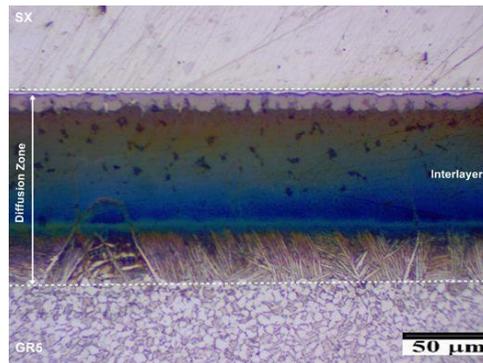


Figure 9. Diffusion zone of welded sample.

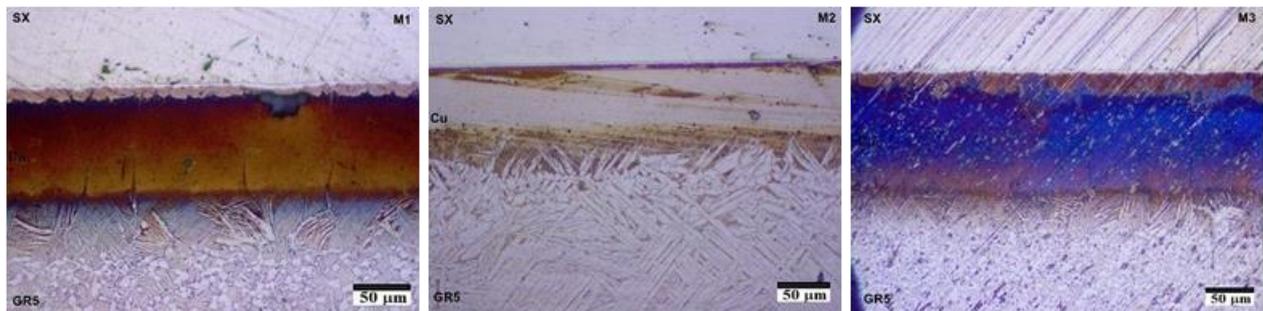


Figure 10. Optical microstructure images of materials M1, M2 and M3

The microstructure image of sample M3 is shown in Figure 11 and Figure 12 of the possible phases resulting from the welding. Based on the EDS analysis result of the

sample, element content is provided in Table 5. According to the analysis results, both the intermediate layer and the combined materials have been diffused mutually.

Table 5. EDS analysis results.

Analysis Points	Ti (at. %)	V (at. %)	Al (at. %)	C (at. %)	Fe (at. %)	Cr (at. %)	Cu (at. %)	Ni (at. %)	Si (at. %)	Possible Phases
1	-	-	-	-	54	18	-	20	8	Fe <sub>17</sub> Ni <sub>6</sub> Cr <sub>6</sub> Si <sub>5</sub>
2	-	-	-	-	55	15	-	20	10	Si <sub>21</sub> Cr <sub>17</sub> Fe <sub>58</sub> Ni <sub>20</sub>
3	-	-	-	2.19	50.9	14.2	12.5	5.35	12.8	Fe <sub>130</sub> Cr <sub>39</sub> Ni <sub>13</sub> Si <sub>65</sub> Cu <sub>28</sub> C <sub>26</sub> Fe <sub>190</sub> Cr <sub>57</sub> Ni <sub>19</sub> Si <sub>95</sub> Cu <sub>41</sub> C <sub>38</sub>
4	-	-	-	-	52,5	14,5	17,5	-	15,5	Fe <sub>17</sub> Cr <sub>5</sub> Si <sub>10</sub> Cu <sub>5</sub>
5	49,61	-	-	-	22,74	5,75	11,34	6,36	4,21	Ti high level
6	70,24	-	5,28	-	8,93	3,83	8,62	3,10	-	Ti high level
7	87,65	7	3,74	1,61	-	-	-	-	-	Ti <sub>41</sub> Al <sub>3</sub> V <sub>3</sub> C <sub>3</sub>
8	94,61	-	-	-	-	-	-	-	-	Ti <sub>188</sub> Al <sub>19</sub>
9	87,49	7,47	5,04	-	-	-	-	-	-	Ti <sub>137</sub> Al <sub>14</sub> V <sub>11</sub>

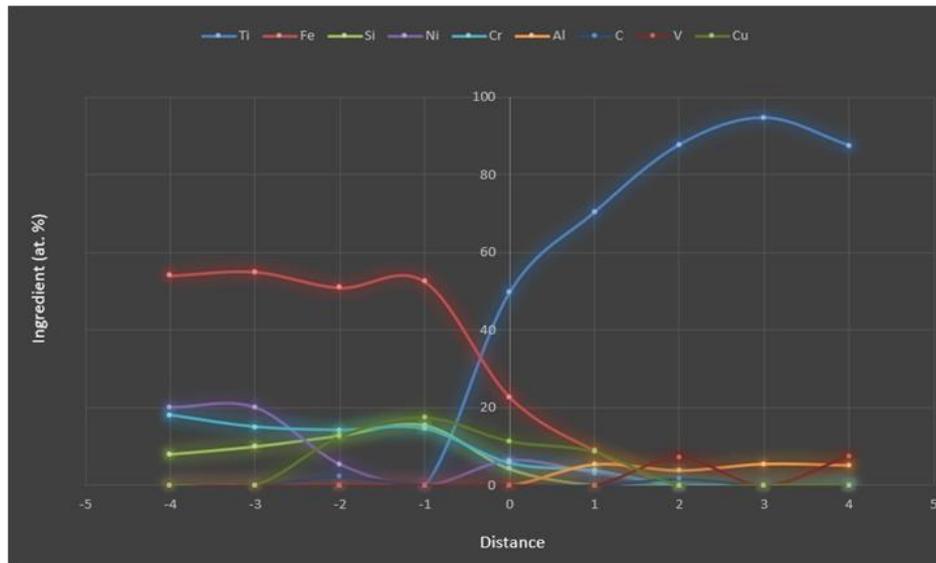


Figure 11. EDS analysis graph.

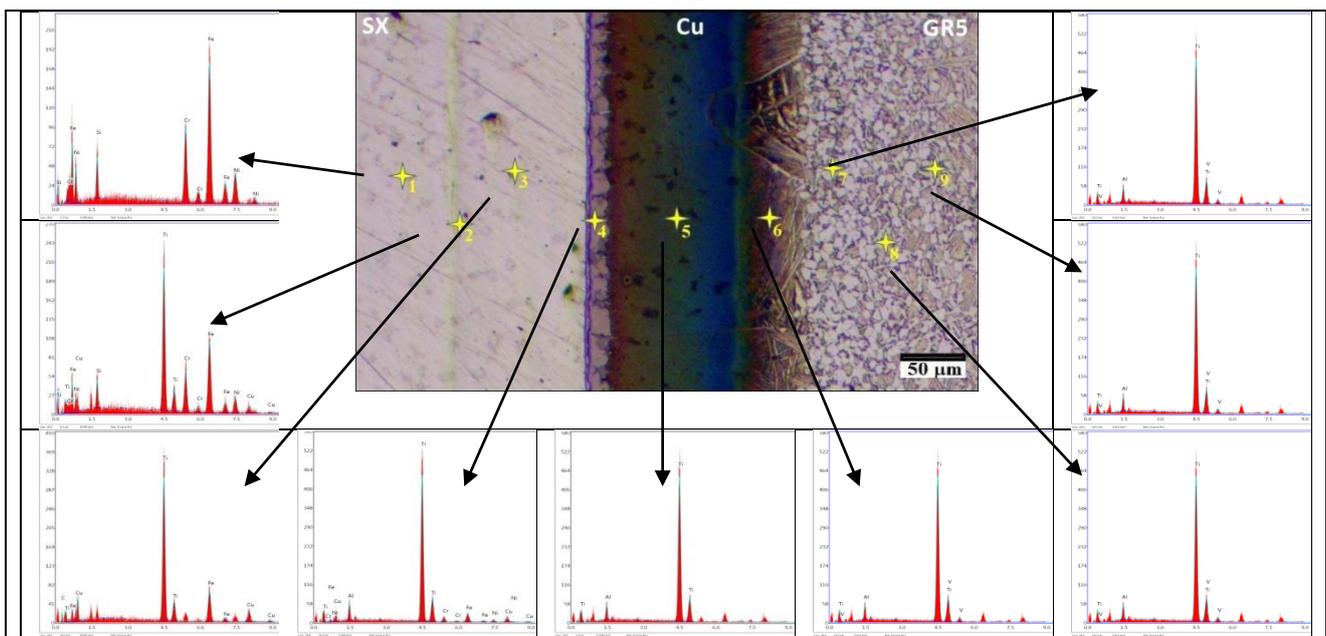


Figure 12. Points and graphics analyzed by EDAX.

When analyzing the EDS analysis of the M3 samples of the most intensive diffusion, around 75 μm on the GR5 side and 50 μm on the SX side, the elemental diffusion is observed. The presence of low carbon in the analysis is thought to have caused the increase in Chrome-Nickel diffusion. Because carbon is a transition element, it has been observed that the increasing welding speed of high rates of carbon can reduce the diffusion of the specified elements.

### Microhardness Results

When the microhardness values of the samples are examined, the hardness of all samples in the diffusion area has higher values than the main materials (Table 6). The hardness has made a peak in the intermediate layer area, then reached the approximate values of the main materials. Possible phases in the intermediate layer zone have allowed the sample to achieve extreme hardness values (Figure 13).

Table 6. Microhardness table.

Material	-40	-30	-20	-10	0	+10	+20	+30	+40
M 1	166.6	305.4	462	655.9	559.8	546.3	453.9	412.7	340.4
M 2	168.9	307.6	316.8	464.4	570.9	573.8	568.1	427	369.4
M 3	183.4	336.4	325.2	368	533.2	476.8	485.6	419.8	405.8

(Vickers hardness value standard ranges for materials: SX: 210-270 HV, GR5: 330-380 HV, Cu: 40-120 HV)

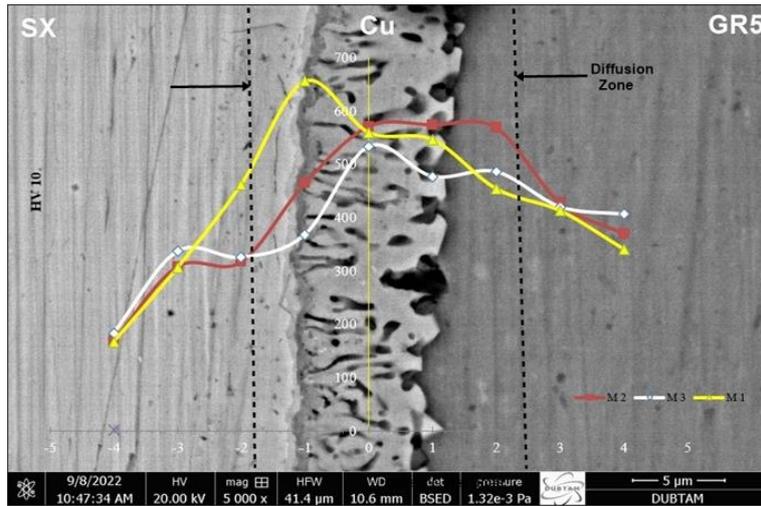


Figure 13. SEM image and hardness graph.

## General Results

The combination of the GR5 titanium alloy and SX steel materials is developed at different temperatures using a copper intermediate layer and the following results have been achieved. In the combination with GR5 and SX copper interface, the diffusion of elements has created a solid solution rich in Fe, Ni, and Ti in the transition zone. This results in higher hardness values in the intermediate area. No microfractures or gaps are identified along the intermediate surface. It may have reduced the possibility of microfractures in the samples during the high plasticity diffusion pressure that is likely to occur on the intermediate surface. Metallographically, the most significant diffusion zone has been found in M3 samples combined at 1020°C. The highest hardness value occurred in M2 samples of 573.8 HV. When examining intermediary surfaces and diffusion zones of welded connections, we have extensively found  $\text{Fe}_{17}\text{Ni}_6\text{Cr}_6\text{Si}_5$ ,  $\text{Si}_{21}\text{Cr}_{17}\text{Fe}_{58}\text{Ni}_{20}$ ,  $\text{Ti}_{41}\text{Al}_3\text{V}_3\text{C}_3$ ,  $\text{Ti}_{137}\text{Al}_{114}\text{V}_{11}$ ,  $\text{Fe}_{17}\text{Cr}_5\text{Si}_{10}\text{Cu}_5$ ,  $\text{Fe}_{130}\text{Cr}_{39}\text{Ni}_{13}\text{Si}_{65}\text{Cu}_{28}\text{C}_{26}$ ,  $\text{Fe}_{190}\text{Cr}_{57}\text{Ni}_{19}\text{Si}_{95}\text{Cu}_{41}\text{C}_{38}$  phases in all samples with the XRD analysis results.

## Ethics committee approval and conflict of interest statement

"There is no need to obtain permission from the ethics committee for the article prepared"

"There is no conflict of interest with any person / institution in the article prepared"

## Authors' Contributions

Güney J: Interpretation of data, drafting of manuscript.

Kejanlı H: supervised the project, critical revision.

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