Microwave joining of SAF 2507 and optimization of elemental constitution using design of experiments

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Abstract. This study focuses on fabrication of SAF2507 similar joints with modified microwave hybrid heating (MHH) method and optimization of the elemental constitution based on energy dispersive spectroscopy (EDS) results. Major input parameters were in this work were filler powder (P), number of graphite rods (R) and process time (T). Each input parameter was varied to three different levels; such as P1, P2 and P3 being no powder, nickel powder and SS304 powder respectively; R1, R2 and R3 being 6, 8 and 10 number of graphite rods respectively; T1, T2 and T3 being 480, 540 and 600 seconds respectively. Experiments were performed as per L9 array based experimental design. EDS based elemental constitution results were used during optimization purpose. Weight percent of Cr, C and Ni have been considered for optimization. Additionally, the mechanical characterization of joints has also been carried out using micro-hardness tests.

Keywords: Analysis of variance; Design of experiments; Energy dispersive spectroscopy; Microwave hybrid heating; Optimization

1 Introduction

Selection of a joining technique depends on various factors such as accuracy, cost-effectiveness, ease of use, environmental impact and energy efficiency. To achieve a healthier and safer work environment with improved processing capabilities, it is necessary to introduce innovative ideas and techniques that can expand the range of materials that can be processed. Joining of materials has become a popular approach for producing both simple and complex parts with good precision and accuracy, but selective and controlled heat flow during joining process remains a significant concern. To address this issue, new joining trends must be explored. One such emerging trend is the joining of various materials using microwave energy. This technique is based on microwave hybrid heating (MHH) and it possesses several benefits such as being non-hazardous, low power consumption, selective and controlled heating etc. Researchers are increasingly fascinated towards this technology due to its potential for joining various types of materials such as

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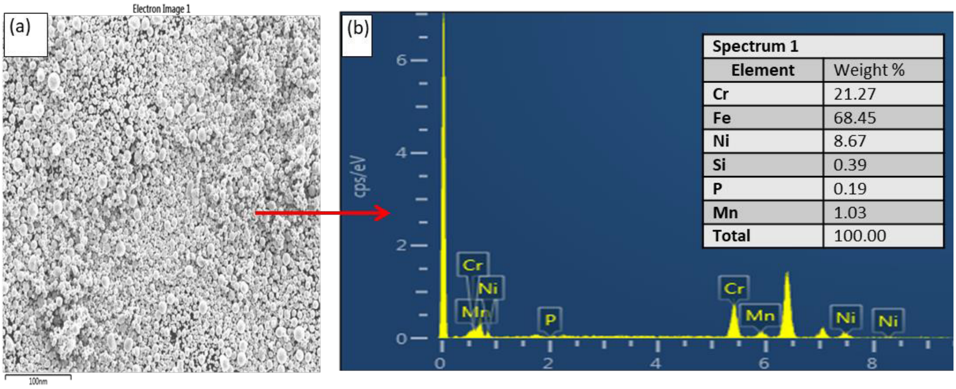
Mild steel [1]–[5], SS304 [6]–[8], SS316 [9], [10], SS2205 [11], Inconel718 [12], [13] , Inconel625 [14]–[18], Cast iron [19], Copper [20], Aluminium [21] etc. Microwave processing enables intensive internal heating that helps in achieving outstanding mechanical properties in shorter processing time as compared to other traditional methods [22]–[30]. Previous investigations have utilized MHH-based joining methods to join a variety of stainless steel specimens [31]. Bagha *et al.* [7] examined SS304 joints prepared using MHH with two different filler powders. They also studied the effects of different filler powder sizes on SS304 weld properties [8]. Bagha *et al.* [6] also successfully joined SS304 in the absence of any filler powder through MHH, which was a remarkable step in reducing the processing cost. Kumar and Sehgal [11] joined SS2205 plates without any filler powder through MHH and observed higher micro hardness at the joint area. Additionally, Kumar and Sehgal [32] joined SS2205 similar plates successfully using MHH with physical and mechanical tests performed on the joints. Bansal *et al.* [9] utilized microwave joining to join SS316 plates with SS316 filler powder and subsequently conducted mechanical tests such as tensile and micro hardness to evaluate the joint's properties. Srinath *et al.* [10] also joined similar material SS316 using MHH with nickel powder and characterized the physical properties of the joint using field-emission Scanning electron microscope (SEM) and X-ray diffraction (XRD) as well as conducting mechanical tests such as tensile strength and hardness. Somani *et al.* [33] examined the joints of SS430 using nickel filler powder with microwave joining and performed SEM, tensile and hardness tests on the joints. Bansal *et al.* [1] conducted the joining of mild steel plates using filler powder of nickel through MHH and characterized the joints using XRD, Electron probe micro-analyzer (EPMA), SEM and micro-hardness tests. Dwivedi and Sharma [2] investigated the effects of various factors on mechanical property tensile strength of joints using MHH. Bansal *et al.* [3] also studied dissimilar joints of MS-SS316 joined through MHH with SS316 filler material characterizing the joints with EDS, micro hardness, tensile and flexural strength tests. Gupta *et al.* [5] proposed a method for joining dissimilar Mild Steel-Stainless Steel plates using microwave joining with nickel filler powder and characterized the joints using XRD, microstructural analysis, tensile strength and hardness tests. Similarly, Srinath *et al.* [4] used microwave joining to join Mild Steel-Stainless Steel plates. An extensive review of the literature shows that, no published work had investigated how various input factors affect Cr, C and Ni elements existence in the joined area of MHH-based joints for SAF2507 joints. This study aims to fill this literature gap and is the key novelty of this effort. To achieve this, the present study produced similar joints and investigated how the number of graphite rods (6, 8, 10), filler materials (no filler, nickel filler, SS304 filler) and process time (480 s, 540 s, 600 s) affect the C, Cr and Ni elements existence in the joints. Optimization results reveal that none of the input factors are significant in affecting the existence of different elements of Cr, C and Ni in joint region. Physical reasoning behind the observations have also been discussed in detail. Mechanical characterization results have also been investigated to ensure proper joint formation.

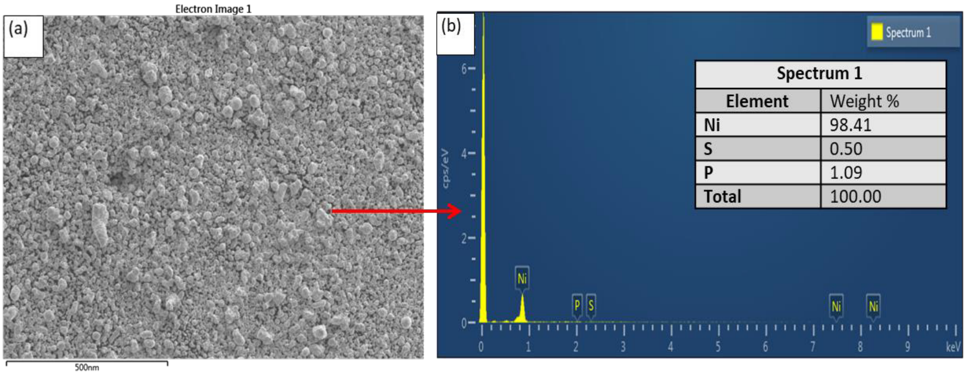
2 Experimental Details

Experiments were performed to join three mm thick flat SAF2507 plates using MHH joining method. SAF2507 specimens were cut to the ASTM E8/E8-09 standard dimensions using wire-cut electric discharge machine. Nine different experimental conditions were used in this work that were based on varying each of the three input parameters to three different levels. First level of the three input parameters were kept as: no filler slurry; six graphite rods; and 480 s process time. During second level, the slurry was formed by mixing nickel powder and epoxy blumer 1450XX; eight graphite rods were used; process time was taken as 540 s. During third level, the slurry was formed using SS304 powder along with epoxy blumer 1450XX; while the number of graphite rods and process time were kept as ten and 600 s respectively. SEM and EDS of filler powders are shown in Figures 1 and 2. Specimen material was placed on ceramic wool and covered with a different ceramic wool piece to shield the specimen material used. Graphite rods were used as a susceptor medium to absorb electromagnetic radiations, providing a path for material heating. Welded joints were cleaned and filed carefully to avoid any foreign particle. The EDS tests of the joints were conducted for examination of the variety of elements existing in the joined area using a set-up available at Metallurgical & Materials Engineering Department, PEC, Chandigarh, India.



Spectrum 1	
Element	Weight %
Cr	21.27
Fe	68.45
Ni	8.67
Si	0.39
P	0.19
Mn	1.03
Total	100.00

Fig. 1. (a) SEM micrgraph of SS304 filler material (b) EDS analysis of SS304 filler material



Spectrum 1	
Element	Weight %
Ni	98.41
S	0.50
P	1.09
Total	100.00

Fig. 2. (a) SEM micrograph of Nickel filler material (b) EDS analysis of Nickel filler material

EDS investigations indicated the presence of several elements and metallic carbides formed in the joint area, which contribute to distinctive metallurgical properties. Figure 3 illustrate the EDS test results conducted at joint area of the SAF2507 specimens that were joined using without any filler powder.Similarily, SAF2507 specimens were joined using nickel and SS304 powder describe in results section.

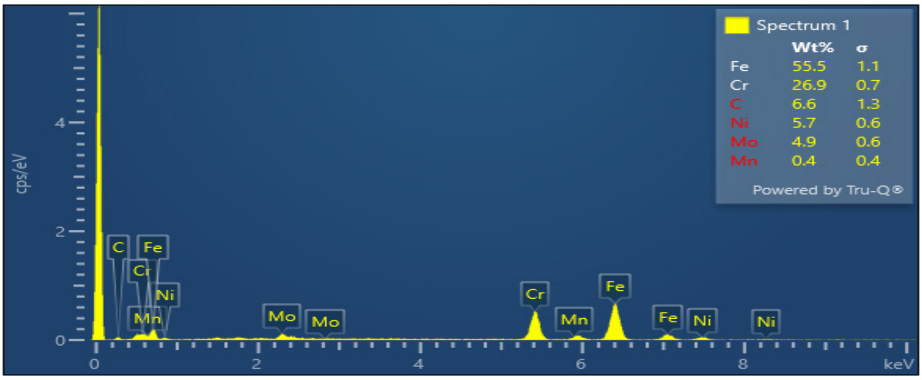


Fig. 3. EDS spectrum of SAF2507 similar joint without filler powder with six graphite rods

By examining Figure 3, it is apparent that the joined area of the SAF2507 joint is characterized by presence of Cr, C and Ni based extreme peaks. The presence of Cr, C and Ni elements in the joined central area signifies the mutual diffusion of the parent materials. The use of graphite rods introduces additional C particles into the joined area and this leads to an increased carbon content at joint region, therefore the EDS results of ten graphite-rods based joints show the highest existence of C content. The founding of hard Cr carbides demonstrated Cr element strong affinity towards C at high temperatures.

3 Results And Discussion

The study aimed to investigate how different types of physical elements affect the joint quality formed by MHH. To achieve this, a Taguchi optimization approach was used specifically on L9 orthogonal array pattern. The study focused on three input process parameters namely filler material, number of graphite rods and process time, which were labelled as different parameters P, R, and T respectively as shown in Table 1. Three levels were chosen for each of these factors and they were no powder (P1), nickel powder (P2) and SS304 powder (P3) for filler material powder (P); six rods (R1), eight rods (R2) and ten rods (R3) for the number of graphite rods (R); and 480 s (T1), 540 s (T2), and 600 s (T3) for process time (T) respectively.

Table 1. Parameters used

	Levels		
Parameters	**I**	**II**	**III**
Filler powder (P)	No powder	Nickel powder	SS304 powder
No. of graphite rods (R)	6	8	10
Process time (s) (T)	480	540	600

Experiments were conducted to investigate composition of elements in the joints formed by MHH. The optimization criteria chosen for this study was "larger the better" for quality physical characteristics. The experimental details were scheduled using an L9 orthogonal array and the outcomes are shown in the Table 2.

Table 2. EDS results show different elements weight percentage

Filler powder (P)	No of graphite rods (R)	Process time (s) (T)	Cr	Ni	C
No powder	6	480	26.9	5.7	6.6
	8	540	29	4.4	7.2
	10	600	29.6	4.9	8.9
Nickel	6	540	23.4	7.5	6.1
	8	600	25	6.7	6.4
	10	480	25.4	6.4	6.6
SS304	6	600	25.5	5.7	5.6
	8	480	24.2	6.2	6.8
	10	540	22.7	5.3	11.9

3.1. Taguchi optimization of Cr content

The investigation based on Taguchi's method was conducted for Cr weight percentage present in the joints, which is illustrated in Table 3. Moreover, Tables 4 and 5 present the corresponding response tables for S/N ratio and mean values of Cr content weight percentage in the joint. Impact of input factors on the S/N ratio and mean of Cr content weight percentage in joint is presented in Figures 4 and 5.

Table 3. Element weight percentage of Cr content in the joint area

Joint name	Filler powder (P)	No of graphite rods (R)	Process time (s) (T)	Cr (Weight %)
J1	No powder	6	480	26.9
J2	No powder	8	540	29
J3	No powder	10	600	29.6
J4	Nickel	6	540	23.4
J5	Nickel	8	600	25
J6	Nickel	10	480	25.4
J7	SS304	6	600	25.5
J8	SS304	8	480	24.2
J9	SS304	10	540	22.7

Table 4. S/N ratio table of Cr content weight percentage in joint area

Levels	Filler powder (P)	No of graphite rods (R)	Process time (s) (T)
1	29.09	28.04	28.12
2	27.81	28.29	27.92
3	27.64	28.21	28.51
Delta	1.45	0.26	0.59
Rank	1	3	2

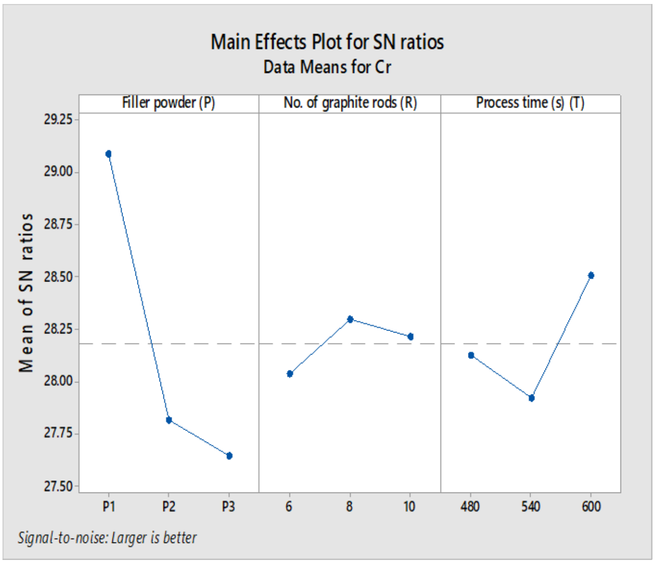


Fig. 4 S/N ratio chart of Cr content weight % in joint area

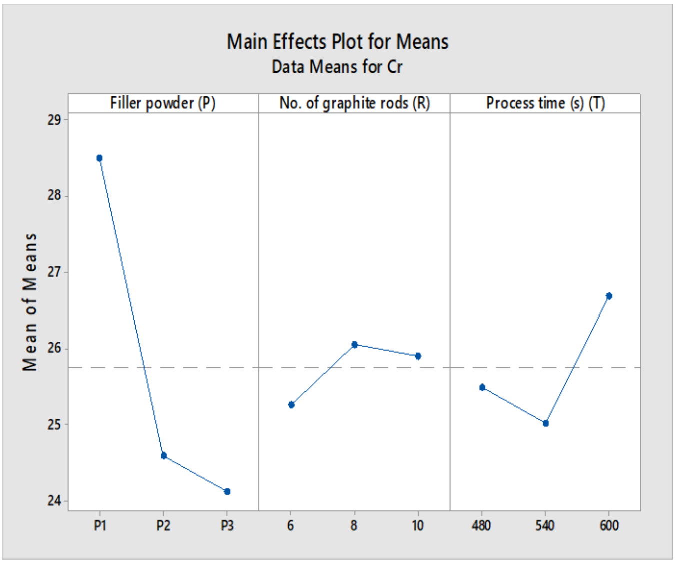


Fig. 5. Mean chart of Cr content weight % in joint area

Table 5. Mean table for Cr content weight percentage in joint area

Level	Filler powder (P)	No of graphite rods (R)	Process time (s) (T)
1	28.50	25.27	25.50
2	24.60	26.07	25.03
3	24.13	25.90	26.70
Delta	4.37	0.80	1.67
Rank	1	3	2

Table 6 displays the results of the ANOVA, indicating that the filler material has the greatest impact on Cr weight %, contributing 76.07%, followed by microwave process time with an impact of 10.82% and no. of graphite rods having the minimum effect on Cr content with an impact of 2.11%. Despite this, the ANOVA investigation outcomes indicate that no significant effect showed by any input factors on the Cr content in the joined area, as their p-values exceed 0.05. The reason for this is that high Cr content already present in the joints.

Table 6. ANOVA chart of Cr weight percentage in the joint area

Source	**DOF**	**Seq SS**	**Adj SS**	**Adj MS**	**F**	**(p< 0.05)**	**Percentage contribution**
Filler powder (P)	2	3.7522	3.7522	1.87611	6.92	0.126	76.07
No of graphite rods (R)	2	0.1043	0.1043	0.05216	0.19	0.839	2.11
Process time (s) (T)	2	0.5336	0.5336	0.26678	0.98	0.504	10.82
Error	2	0.5424	0.5424	0.27119			11
Total	8	4.9325					

3.2. Taguchi Optimization of Ni Content

Table 7 displays the results of Taguchi's analysis conducted for Ni content weight % in the joints. Tables 8 and 9 demonstrate the response chart for S/N ratio and mean of Ni content weight % in the joint respectively. Additionally, impact of input factors on the S/N ratio and mean for Ni content weight % in joint is presented in Figures 6 and 7.

Table 7. Element weight percentage of Ni content in the joint area

Joint name	**Filler powder (P)**	**No of graphite rods (R)**	**Process time (s) (T)**	**Ni (Weight %)**
J1	No powder	6	480	5.7
J2	No powder	8	540	4.4
J3	No powder	10	600	4.9
J4	Nickel	6	540	7.5
J5	Nickel	8	600	6.7
J6	Nickel	10	480	6.4
J7	SS304	6	600	5.7
J8	SS304	8	480	6.2
J9	SS304	10	540	5.3

Table 8. S/N ratio table of Ni content weight percentage in joint area

Levels	**Filler powder (P)**	**No of graphite rods (R)**	**Process time (s) (T)**
1	13.93	15.91	15.70
2	16.72	15.08	14.95
3	15.15	14.80	15.15
Delta	2.79	1.11	0.74
Rank	1	2	3

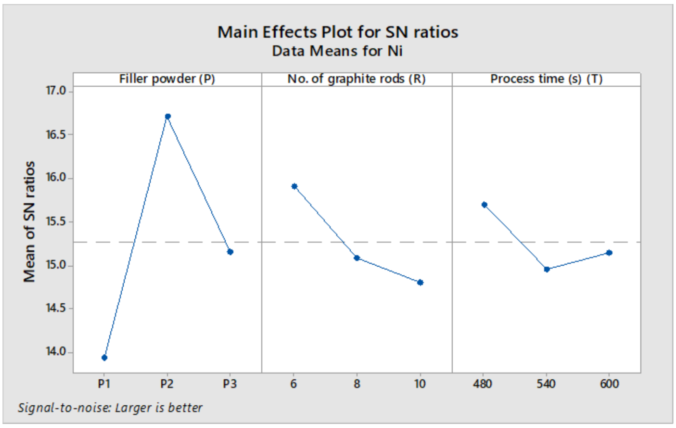


Fig. 6. S/N ratio chart of Ni content weight % in joint area

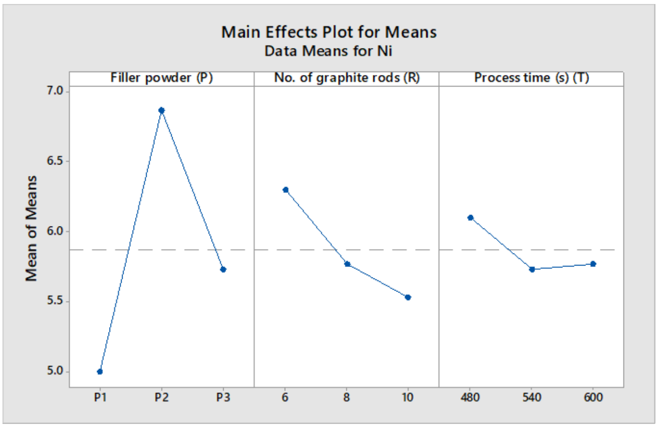


Fig. 7. mean chart of Ni content weight % in joint area

Table 9 Mean table of Ni content weight percentage in joint area

Level	Filler powder (P)	No of graphite rods (R)	Process time (s) (T)
1	5.000	6.300	6.100
2	6.867	5.767	5.733
3	5.733	5.533	5.767
Delta	1.867	0.767	0.367
Rank	1	2	3

Table 10 displays the ANOVA outcomes, indicating that parameter filler material has the highest impact on Ni weight %, contributing 72.28%, followed by no. of graphite rods with a contribution of 12.33% and microwave process time having the minimum effect on Ni content with an impact of 5.52%. Despite this, the ANOVA investigation outcomes indicate that no significant effect showed by any input factors on the Ni content in the joined area, as their p-values exceed 0.05. The reason for this is that small amount of filler powder used as compared to Ni content already present in the joints.

Table 10. ANOVA chart of Ni weight percentage in joint area

Source	**DOF**	**Seq SS**	**Adj SS**	**Adj MS**	**F**	**(p< 0.05)**	**Percentage contribution**
Filler powder (P)	2	11.6962	11.6962	5.8481	7.32	0.120	72.28
No of graphite rods (R)	2	1.9960	1.9960	0.9980	1.25	0.444	12.33
Process time (s) (T)	2	0.8934	0.8934	0.4467	0.56	0.641	5.52
Error	2	1.5971	1.5971	0.7986			9.87
Total	8	16.1828					

3.3. Taguchi Optimization of C Content

The investigation based on Taguchi's method was conducted for C weight percentage present in the joints, which is illustrated in Table 11. Moreover, Tables 12 and 13 present the corresponding response tables for S/N ratio and mean values of C content weigh % in the joint. Impact of input factors on the S/N ratio and mean of C content weight % in joint is presented in Figures 8 and 9.

Table 11. Element weight percentage of C content in the joint area

Weld name	**Filler powder (P)**	**No of graphite rods (R)**	**Process time (s) (T)**	**C (Weight %)**
J1	No powder	6	480	6.6
J2	No powder	8	540	7.2
J3	No powder	10	600	8.9
J4	Nickel	6	540	6.1
J5	Nickel	8	600	6.4
J6	Nickel	10	480	6.6
J7	SS304	6	600	5.6
J8	SS304	8	480	6.8
J9	SS304	10	540	11.9

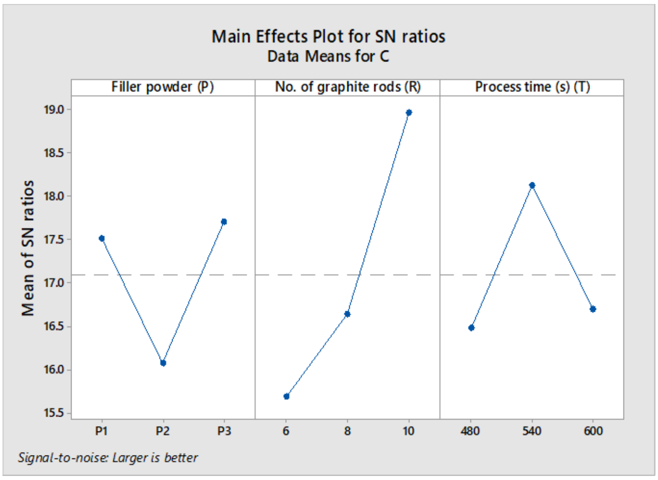


Fig. 8 S/N ratio chart of C content weight % in joint area

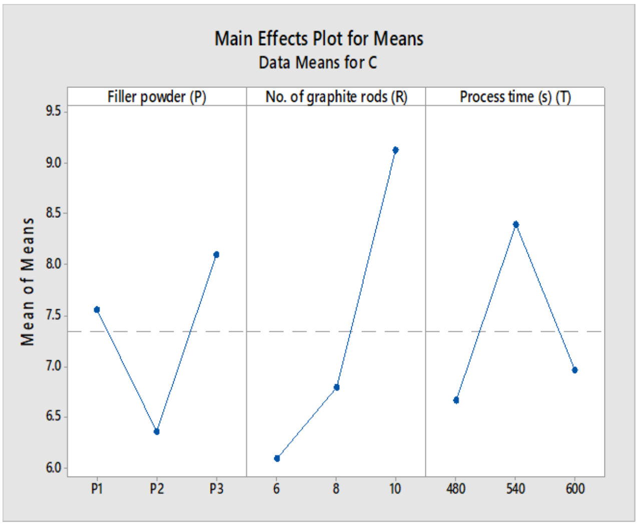


Fig. 9 mean chart of C content weight % in joint area

Table 12. S/N ratio table of C content weight percentage in joint area

Level	Filler powder (P)	No of graphite rods (R)	Process time (s) (T)
1	17.51	15.69	16.48
2	16.07	16.64	18.12
3	17.71	18.96	16.69
Delta	1.63	3.28	1.64
Rank	3	1	2

Table 13. Mean table of C content weight percentage in joint area

Level	Filler powder (P)	No of graphite rods (R)	Process time (s) (T)
1	7.567	6.100	6.667
2	6.367	6.800	8.400
3	8.100	9.133	6.967
Delta	1.733	3.033	1.733
Rank	3	1	2

Additionally, Table 14 displays the ANOVA outcomes for C weight %, indicating no. of graphite rods is the most influential parameter, contributing 53.77%, followed by microwave process time with an impact of 15.13% and filler material having the minimum effect on C content with an impact of 15.05%. However, the ANOVA investigation outcomes indicate that no significant effect showed by any input factors on the C content in the joined area, as their p-values exceed 0.05.

Table 14. ANOVA chart of C weight percentage in joint area

Source	DOF	Seq SS	Adj SS	Adj MS	F	(p< 0.05)	Percentage contribution
Filler powder (P)	2	4.770	4.770	2.385	0.94	0.516	15.05
No of graphite rods (R)	2	17.038	17.038	8.519	3.35	0.230	53.77
Process time (s) (T)	2	4.793	4.793	2.396	0.94	0.515	15.13
Error	2	5.087	5.087	2.544			16.05
Total	8	31.689					

4 Micro-hardness results

The mechanical characterization of the joint was conducting using micro-hardness tests at various positions. To determine the hardness values of the joint, a Vickers hardness tester was used with 1 kg load applied for 10 seconds. The results revealed that the hardness of the joint area was higher in comparison to the parent alloy region. Figure 10 displays the average micro-hardness values obtained at different positions of the joints. It was discovered that the higher hardness in the joint area in comparison to parent alloy region was attributed to carbon absorption from the susceptor rods.

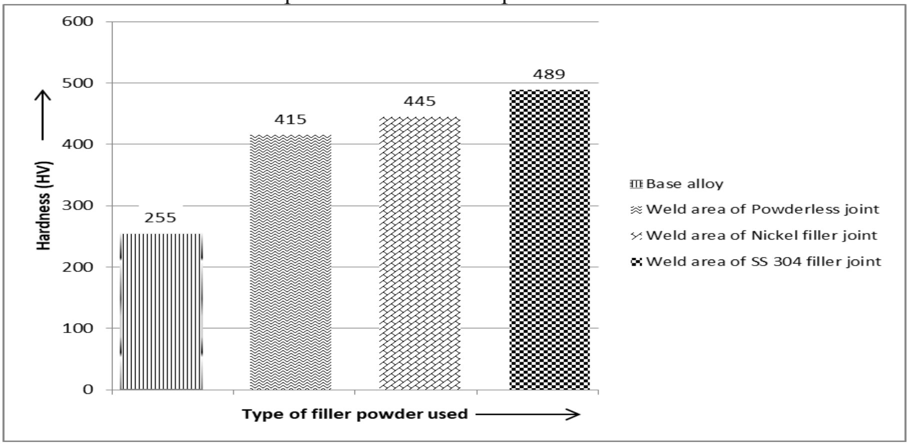


Fig 10. Comparison of microhardness of joints with different filler powders formed through MHH.

5 Conclusions

The paper investigated the microwave joining of SAF2507 plates using three different parameters: filler powder, number of graphite rods and process time. L9 orthogonal array was selected for investigation and EDS was employed to perform physical characterizations of the joints to determine the weight % of different types of elements such as Cr, C and Ni in the final joint formed. Filler powder was not found to be possessing any significant effect on element constitution because of it being used in a very small quantity which is even lesser than total mass of the joined specimens. Further, the graphite rods presence helped in improving the C content in joint region thereby increasing the hardness of the joint area. But due to the small range of graphite rods considered in this work, which varied between six to ten only, the effect of such rods on elemental composition was also not found to be significant. Similarly the process time does not affect significantly the elemental constitution due to the reason that variation in process time does not have a strong impact on metallurgical diffusion process. The hardness of the joined area was determined to be significantly larger in comparison to the base alloy area, primarily because of carbon absorption from the utilized susceptor.

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