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MICROWAVE HEATING OF COMPOSITE MATERIAL REINFORCED WITH NATURAL CERAMICS – TECHNICAL AND ECONOMICAL ASPECTS

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***Abstract:** The present paper proposes the realization of some researches related to microwave heating of composite material based on Fe97.5%Gr0.5%Cu2% reinforced with natural ceramic Rapana Thomasiana powders for improvement of preheating conditions of composites that are subject to welding. The study has been focused on microwave heating mechanism for low power injection during sintering process of composite material in order to obtain economic efficiency of the process in terms of sintering time, energy saving and low carbon footprint. The results showed that, for 10% of Rapana Thomasiana in chemical composition of the samples, the thermal treatment process is feasible for microwave heating. The targeted temperatures were 350 °C and 950 °C and these values were reached after 75 s and 700 s. In addition, the process proved to be very cheap in terms of staff costs and energy consumption.*

***Key words:** composite material, microwave heating, economic efficiency.*

1. INTRODUCTION

The welding of steel materials usually requires a preheating of the base materials in order to reduce the hydrogen diffusion rate in materials at ambient temperature. This process will reduce the risk of cracking and therefore will lead to the improvement of quality of the welds. The preheating of base materials is used to reduce the cooling rate in base materials as well as in welds.

Young et al. [1] reported that preheating before welding raised the γ content of the fusion zone and improved the impact toughness. Padhy et al. [2] studied the influence of temperature on hydrogen reduction using preheating and post-heating of welds. They concluded that both treatment process is more efficient, in terms of energy saving and therefore more economic, when the preheating or post-heating is applied at low temperature. Yurioka [3] studied several preheating methods for structural steels, from a mild steel and TS780 grade steel with carbon contents between 0.034% and 0.234%C. The

conclusion was that all methods will avoid the heat affected zone (HAZ) cold cracking. Chakraborty et al. [4] studied the assisted cracking susceptibility of modified 9Cr-1Mo steel welds in order to determine lower critical stress for different preheating and combination of pre- and post-heating conditions. In the study, the authors reported that for both treatment methods, the diffusible hydrogen content of welds has been reduced to 1.17 mL/100g from 4.7 mL/100g.

The preheating and post-heating of welds requires additional procedures that lead to higher processing costs and therefore to low economic efficiency. By combining the thermal treatment with welding process in the same time, the reduction of diffusible hydrogen will be done without any additional processes. However, this approach requires additional thermal source that should be complementary and synergic with the welding process. Microwave technology is a non-contact heating source that creates heat directly in base materials. The interaction between microwaves and metals, normally leads

to arc discharge, but the technology can be applied as an energy efficient tool for a number of processes. Gupta et al. [5] studied the heating mechanism for sintering, brazing, carburizing and annealing of metals in microwave field. They presented different methods for heating using microwaves as well as necessary technologies and equipments that can be used in microwave processing of metals. Yoshikawa [6] studied the interaction between microwaves and materials focusing on discussions related to boundary conditions of electromagnetic field on metal surface in terms of surface impedance. They reported to what extent the microstructure is altered by microwave heating. Sun et al. [7] researched the microwave – matter interaction focused on microwave heating mechanism and temperature profile within the sample for both microwave heating and microwave hybrid heating. Microwave processing requires customized technical solutions for each heating process. Sintering, welding, melting and any other process that uses microwave heating, need particular approach in terms of applicator design, reaction chamber design, injected power, matching load impedance and other specific parameters. Colombini et al. [8] researched a new hybrid microwave and induction heating applicator for metal making. They used numerical simulation to design and optimize the microwave and hybrid microwave-induction units for simulation the influence of the shifting microwave generator frequency band on heat generation. Other researchers focused their studies on microwave heating of metal oxides substrate composites. Ano et al. [9] reported that oxide ceramic substrates such as Al_2O_3 , TiO_2 , MgAl_2O_4 or SrTiO_3 contribute to the changing relative permittivity and conductivity of the composite material. The influence of oxide ceramic substrates on temperature distribution as microwave heating effect contributed to the decreasing of microwave injected power and heating time. Rapana Thomasiana (RT) seashell contain more than 95% CaCO_3 being mostly used in bone implants [10, 11]. According to Savu et al., powders based on RT can be heated in microwave field in order to obtain different products from natural wastes [12]. In addition, RT powders can be combined with polymers sheets for 3D printing applications [13-15].

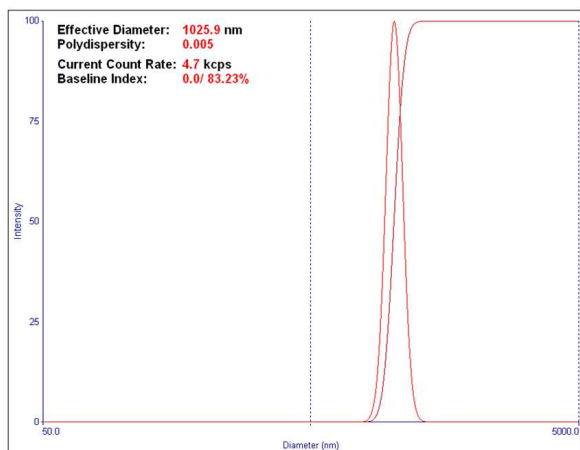
Microwave heating represents a faster technology for processing materials, but it is well known that the process is unstable. Savu et al. [16, 17] studied microwave heating mechanism for sintering of ceramic materials. Also, several researches were reported in the field of microwave heating of cordierite material. In all cases, the researchers outlined two unwanted phenomena: thermal runaway and microwave arc discharge in free atmosphere. Researchers reported that barium ferrites suffered thermal runaway at low microwave injected power. For 600 W injected power, the temperature of sample increased fast from 620 °C to 975 °C in less than 10 s. The samples have been damaged due to the microwave plasma arc initiation [18, 19].

Microwave heating can contribute to the fast preheating and post-heating of base materials that are welded using electric arc processes. However, metal materials reflect microwaves and no heat will be developed. By reinforcing the metal materials with ceramics, the composite materials will convert the microwaves into heat.

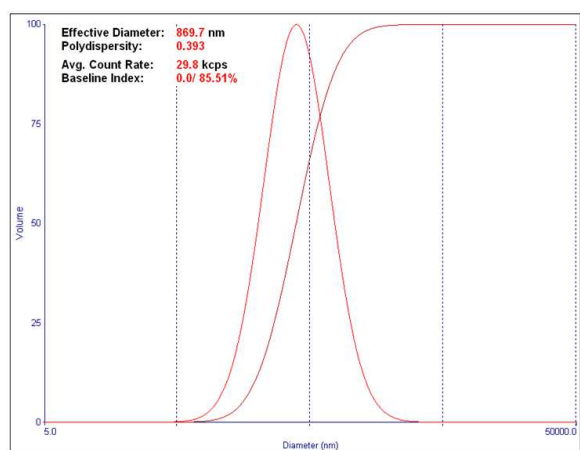
This paper aims to introduce natural ceramic materials from RT seashells into composites nanostructured powders based on Fe97.5%Gr0.5%Cu2%. The reinforcement of metal composite powders with RT is expected to lead to a better conversion of microwave energy into heat, and therefore, to the better elimination of hydrogen through preheating of base materials in the same time with welding process. In addition, in this study, will be presented calculations related to energy consumption and staff costs, in order to determine the economic efficiency of the microwave heating.

2. MATERIALS AND METHODS

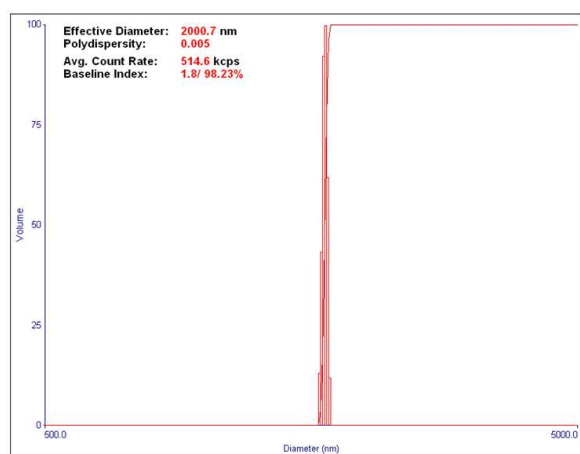
The materials used in experimental program were Fe100% (code S1) powders, mechanical alloyed composites powders based on Fe97.5%Gr0.5%Cu2% (code S2) and RT powders (code S3). Before homogenization, the powders were analyzed using Brookhaven 90 Particle Sizer Analyzer (Brookhaven Instruments Corporation, USA, 2008). The effective diameter size for each sample tested are presented in Figure 1.



a.



b.



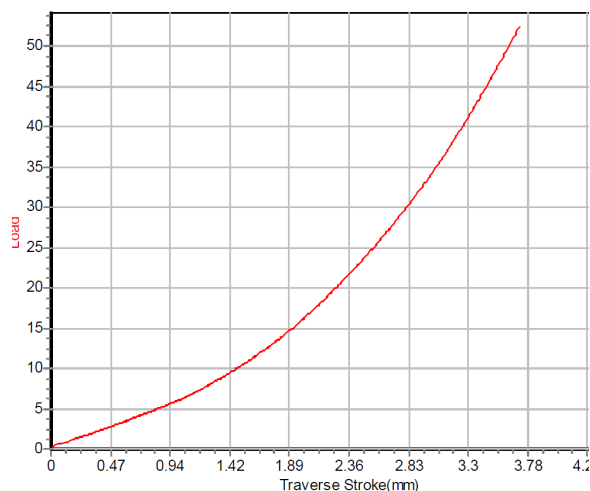
c.

Fig. 1 Grain size distribution of initial powders
 a. S1, b. S2, c. S3

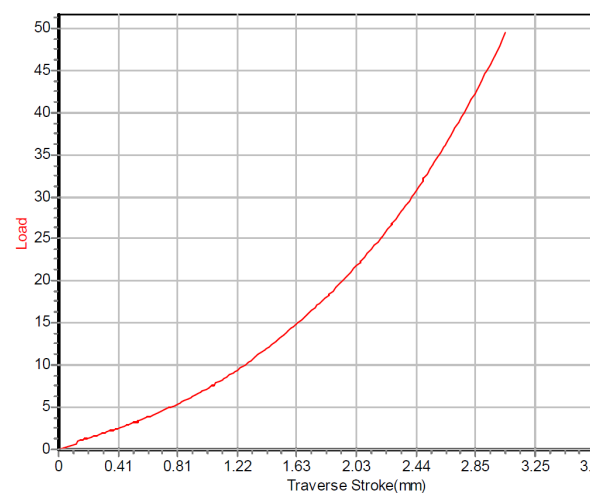
According to the results obtained from distribution size analysis, the effective diameter of S1 was 1025.9 nm, for initial alloy S2 was 869.7 nm and for the S3, the grain size was

2000.7 nm. The initial powders have been pressed using a cylinder die having inner diameter 15 mm using a pressing force equal with 300 MPa. The device used for pressing process was LBG Universal Testing Machine (LBG srl, Italy, 2010). The homogenous mixture S2(90%) + S3(10%) has been obtained after a mixing process in Pulverisette 6 planetary ball mill (Fritsch, Nitech srl, Romania, 2009). The samples have been obtained by processing the powders with the following parameters:

- milling speed 150 rot/min with changing rotational direction once at 5 min.
- the grinding balls from steel had the diameter 10 mm.
- the milling process has been realized in dry environment for 20 min.



a.



b.

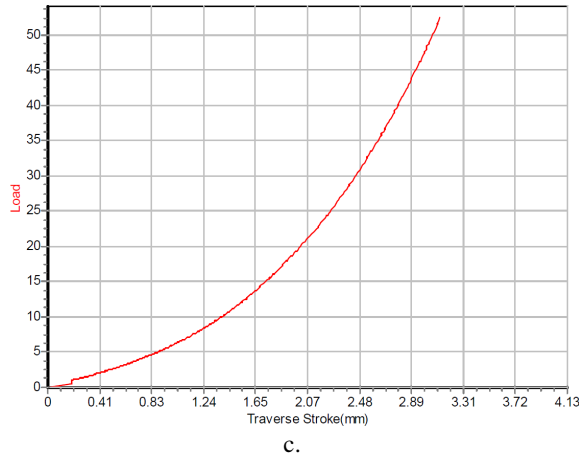


Fig. 2 Pressing curves for initial powders
a. S1, b. S2, c. S2+S3

Figure 2 presents the results of compression process for initial powders and homogenous mixture. Table 2 presents the characteristics of the samples before sintering in conventional sintering furnace.

Table 1
Characteristics of the crude samples after pressing

Code	Diameter [mm]	Height [mm]	Weight [g]
S1	15.18	3.65	4.00
S2	15.20	3.30	3.84
S2+S3	15.19	3.70	3.77

The samples have been sintered in conventional microwave oven at 1100-1300 °C. After the sintering of S1, S2 and S2+S3 samples in conventional oven, the samples were subject of microwave heating in order to establish to what extent the presence of natural ceramic (Rapana Thomasiana) influences the performance of heating process. The microwave heating has been applied using a 6000 W microwave generator (Muegge GmbH, Germany, 2020), a rectangular waveguide type WR340 and an automatic tuner for matching load impedance (SK-Team, Slovakia, 2009). The software for recording the data from matching the impedance was HomSoft (SK-Team, Slovakia, 2018). The cooling of the waveguide has been done using two cooling flanges located at the beginning and the end of waveguide. The cooling of magnetron head has been realized using open cooling circuit having the water pressure between 2 and 4 bar. The heating chamber has been designed to fit to

the rectangular waveguide and also to allow the measurement of the temperature on sample' surface. The temperature has been measured using an infrared pyrometer CT Glass (Optris GmbH, Germany, 2009) with range between 250 and 1650 °C. The software used for temperature recording was CompactConnect (Optris GmbH, Germany, 2018). Figure 3 presents the experimental installation for microwave heating of samples.

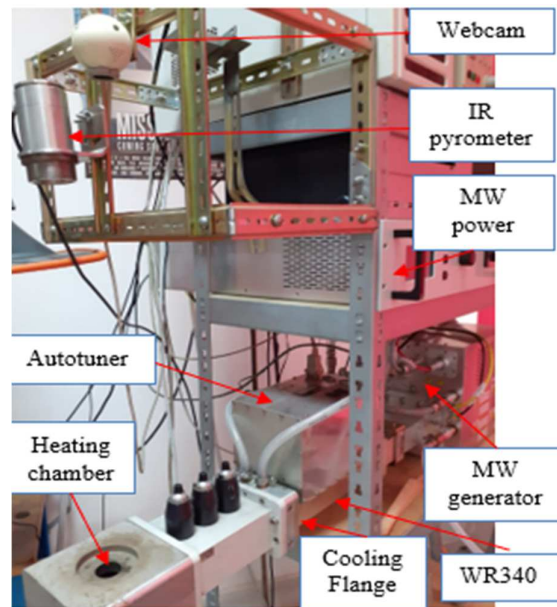


Fig. 3 Experimental microwave heating installation

The samples were placed in a ceramic crucible similar with ceramic supports used in arc welding for retaining and avoiding the flow of melted material. Figure 4 presents the heating chamber with S1 sample and a snapshot from matching load impedance tuning process.

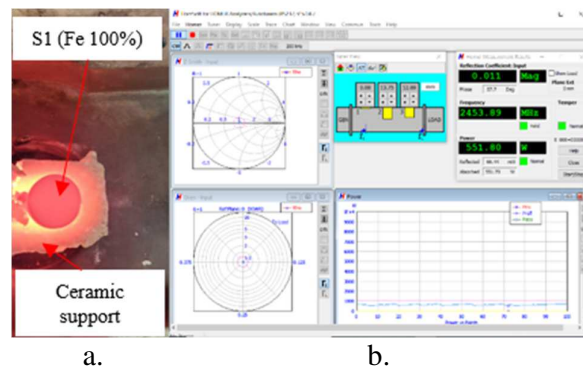


Fig. 4 Capture obtained during microwave heating
a. sample in heating chamber, b. automatic matching load impedance

The microwave heating started with 600 W and the targeted temperatures were 350 °C and 950 °C, taking into consideration the heat treatments used for reduction of hydrogen before, during and after welding processes. The output microwave power has been adjusted from 600 W to 1200 W, for each sample in particular, in order to ensure a smooth and continuous increase of temperature.

3. RESULTS AND DISCUSSIONS

The process started with high increase of temperatures for all three samples taken into consideration. However, the high percentage of the Fe in composition of samples, lead to plasma arc initiation at the beginning of the heating process until the automatic matching load tuner has calculated the optimal electric circuit, and the transfer of the microwave power from generator to load was maximum. In addition, the reflected wave decreased to less than 23.4 W.

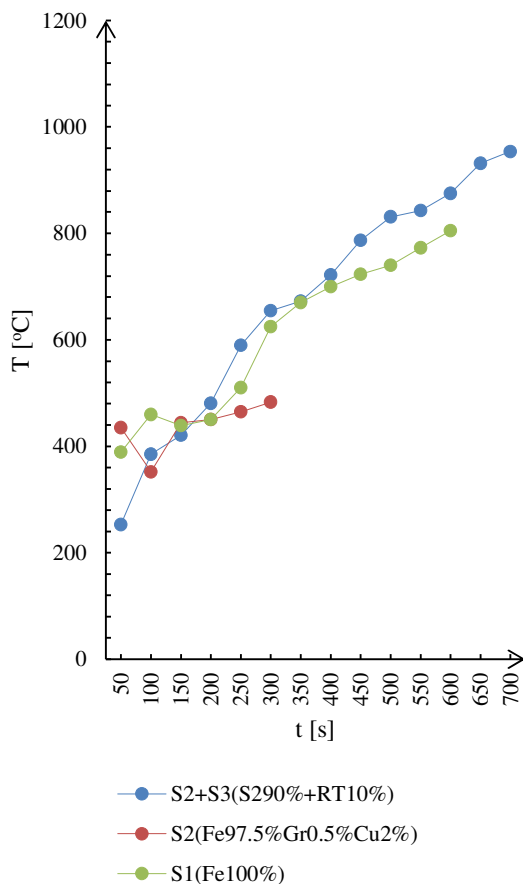


Fig. 5 Temperature evolution during microwave heating

The temperature of sample S1 increased constantly after 300 s, but the maximum value of the temperature was 805 °C not enough for hydrogen reduction in welds. Similar increase, but with better results, has been recorded for composite S2 with 10% Rapana Thomasiana. The presence of ceramic material helped the process to be more stable and the maximum temperature was reached after 700 s. The process has been stopped when the temperature was 954 °C. The third sample (S2) did not heat very well, especially due to the presence of copper in chemical composition. The process proved to be highly unstable and the plasma arc was initiated. The process has been stopped after 300 s due to process instability which affected and damaged the sample.

Regarding the economic aspects of the microwave heating of samples, is well known that being a faster process, the costs for processing are lower. In this study, the economical efficiency has been calculated taking into account the following criteria:

- electrical energy consumption by the process itself
- electrical energy consumption by the auxiliary devices such as computer process, heating regulator
- direct expenditures for personnel in a certain period of time

The electrical energy absorbed during microwave heating ramp can be calculated with the following equation:

$$E_{MW} = \sum_{i=1}^3 E_i \quad (1)$$

where E_i [Wh] represent the consumption of each device that contribute to the microwave heating. The consumption was measured using Digital Wattmeter CA 6520 (Chauvin Arnoux, France, 2010) for microwave heating period.

$$E_i = \sum_{i=1}^3 P_i \cdot t_{process} \quad (2)$$

where P_i [W] have been measured during microwave heating process. In addition, has been performed an evaluation of staff costs for the personnel involved in the heating process.

$$C_{staff} = \frac{\sum DC}{t_{month}} \cdot t_{process} \quad (3)$$

where C_{staff} represents the total costs with staff for microwave heating process for one product, DC represents total direct costs and $t_{process}$ represents the total time for one microwave heating process. Table 2 presents the evaluation of costs for microwave heating process taking into account the medium gross salary of a technician.

Table 2

Total costs for microwave heating			
Process	Technology [EUR]	Staff [EUR]	Total [EUR]
MW	0.15	0.18	0.33

The total gross salary was considered to be 500 euro/month and the price of the electrical energy was taken as 0.2 euro/kWh. The total time of the microwave heating process was considered to be 700 s which has been obtained for S2 sample combined with Rapana Thomasiana.

4. CONCLUSION

Microwave technology can be applied for preheating or post-heating of composite materials having metal matrix reinforced with natural ceramics obtained from Rapana Thomasiana seashells.

The differences between all samples tested in microwave field were not high, in terms of temperature evolution in time, but only the composite material reinforced with natural ceramic, succeeded to reach the temperature required by heat treatment for hydrogen reduction.

The target temperature, around 950 °C, required by preheating and post-heating treatment has been reached after 700 s only with tuning of load impedance. Without matching load tuner, the process presented high instability and the unwanted phenomenon like thermal runaway occurs from the very beginning. However, even with matching load impedance, the composite material S2 (Fe97.5%Gr0.5%Cu2%) was impossible to be stabilized due to the presence of high conductive copper in chemical composition. On the other hand, the

sample S1 (Fe100%) had good behavior in microwave field, in terms of thermal runaway and microwave plasma arc, but the maximum temperature did not exceed 805 °C. The best results were obtained for composite materials with metal matrix reinforced with natural ceramic obtained from Rapana Thomasiana seashells. Similar with previous cases presented above, the process needs matching load impedance. However, the loss power, which was reflected, has been lower comparing with composite materials without reinforcement with natural ceramic. This can be explained by the microwave heating mechanism where the presence of ceramics contributes to the improvement of absorption and conversion of microwaves into heat.

Regarding the economic efficiency of the microwave heating for preheating and post-heating of the samples subjected for welding, the total cost of processing is a cheap one comparing with others. Taking into consideration reduced time for heating, costs with staff and electrical energy, the microwave heating needs a total cost less than 0.33 euro for one product.

5. ACKNOWLEDGEMENT

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Încălzirea cu microunde a materialelor composite ranforsate cu ceramic naturală – aspecte tehnice și economice

Lucrarea de față propune realizarea unor cercetări privind influența ceramicii naturale de ranforsare a matricii unui compozit pe bază de Fier, Cupru și Grafite asupra capacității de absorbție și conversie a undelor electromagnetice de înaltă frecvență în căldură. Cercetările au fost efectuate pentru îmbunătățirea condițiilor de reducere a hidrogenului ce poate conduce la fisurarea îmbinărilor sudate cu arc electric. Au fost studiate elemente privind stabilitatea procesului de încălzire, evitarea fenomenului de ambalare termică respectiv de apariție a arcului de plasma de microunde, precum și a eficienței economice a procesului de încălzire cu microunde pentru tratamentul termic al materialelor de bază înainte și după efectuarea îmbinării

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