RESEARCH ON SOLAR SINTERING FOR ELECTRICAL CONTACTS

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ABSTRACT: Solar sintering is a promising method based on clean energy that could be used for obtaining electrical contacts. As shown in this paper, in terms of thermal stability, it is better than other modern sintering technologies like microwave and spark plasma sintering that have applications in the electrical field. This paper analyzes the possibility to sinter the copper alloy in order to improve its properties without decreasing the electrical conductivity of the copper material. The compacted pellets of Cu-TiC-graphite were sintered in a solar furnace by heating and maintaining the pellets at a precise temperature in a controlled environment (Argon) at temperatures around 700-850°C for a maintain time of 20-30 minutes. This configuration was performed by using the MSSF Solar Furnace of the PROMES-CNRS facility in France. Initial tests have shown that electrical conductivity does not suffer a significant reduction while wear rate and friction coefficient are better or similar to those for pure copper

KEY WORDS: solar sintering, electrical contacts, copper alloys, thermal stability, clean energy

1. INTRODUCTION

Recent and previous works using the MSSF solar furnace of the PROMES-CNRS facility from Font-Romeu in Southern France have demonstrated that it is possible to use concentrated solar radiation as a sustainable green heat source for producing materials with improved properties from copper and other metal powders, and especially electrical for copper-based materials [1, 2].

Our solar sintering experiments for copper-based powder mixture are to reveal that the mechanical and electrical properties of the sintered copper-based material are almost comparable to those of the counterpart prepared through other sintering processes: microwave [3] and spark plasma sintering [4]. Compared to these, solar furnace sintering should be further pursued by experimental verifications for other materials with similar properties because this ecological, sustainable, cheap, and quick method would raise productivity of copper-based alloys with acceptable quality for electrical applications, like electrical contacts, at reasonable price with minimized environmental load.

In the present study, Cu-TiC-graphite compacted pallets were sintered at temperatures around 700 - 850 ^{o}C in the solar furnace. As in most cases, compacted powder mixtures consisting of metal and graphite are enclosed in a controlled environment with gas for a standard reaction time of 30 min [5]. In order to obtain the optimized Cu-TiC-graphite alloy different powder ratios, compacting force and different solar heat sintering cycles were used.

Figure 1. Sintered probe

2. COPPER-BASED ALLOYS FOR ELECTRICAL CONTACTS

Due to its excellent thermal conductivity, electric properties, resistance to the corrosion, and good mechanical properties, copper has different applications in the modern society: connectors, contact switches, radiators for automobiles, elements of thermostats, etc. [6]. Nowadays there is an increase in the demand for materials with high strength, good electrical and thermal conductibility and corrosion resistance. Moreover, cooper is the best choice for electrical contacts used in antennas and advanced telecom systems due to the increased electrical efficiency with benefits in increasing RF energy. Copper has many advantages and is used as contact material or layer in almost all electrical contact and micro-contacts applications but it is characterized by low yield strength and wear resistance [7]. Electrical contacts used in power switching rely on composites of copper [8, 9]. Copper can be used with addition of alloy elements to increase the main properties [10]. Contacts and structural parts can be manufactured through the process of powder metallurgy, which can reduce or eliminate costly machining processes and allow less scrap loss, compared to other forming methods [6].

2.1 Sintering issues

A degradation mechanism caused by oxidation, fretting and thermal expansion or due to the arching phenomena that cause welding and local melting can appear at every contact's open/closed loop. Therefore, it is necessary to improve the performance of the cooper alloy and the usual method is to add supplementary elements or strengthen with a solid solution that will result in the decrease of electrical conductivity of the cooper alloy. Further improvement of cooper alloys can be achieved by using supplementary reinforcing particles combined with the sintering process. Wang et al. [11] by using the reaction of graphite and soluble Ti obtained in-situ the TiC as reinforcement material for the cooper matrix. Ultrafine structured Cu–TiC composites were also fabricated by [12] using the mechanical milling procedure. Good results were reported in terms of electrical conductivity at an optimized pressure temperature sintering ratio of 800 °C/ 60 Mpa. In a similar work, [13] fabricated ultrafine-grained copper alloys with nanoscale TiC particles by mechanical milling and spark plasma sintering [14].

An efficient tool for grinding and mixing many materials into fine powder is presented in [15], in which a copper-graphite composite powder was successfully fabricated by ball milling technique. An overview of the benefits of sintering composite materials based on Cu, TiC and graphite and their applications is given in [16]. Some of the benefits include oxide reduction and improved sintering properties such as: strength, hardness, electrical conductivity, density, ductility and deformation response. Another aspect concerns wettability, as depicted in [17]. Since graphite and TiC have poor wettability with cooper melts, the melting-casting in situ synthesis method is not recommended. Therefore, it is necessary to develop a suitable method for fabricating a Cu alloy based on TiC and graphite. Moreover, the authors conclude that graphite is an ideal candidate for the production of different kinds of contacts, conductors, particles and other sintered articles for the conduction of electric current in various systems due to its physical-mechanical and electrical properties, while the authors insist on a highly conductive material with improved strength based on heat treated copper alloys (Cu-Ti and Cu-Ti-Tisi) [7]. Other authors, tackle sintering issues such as porosity, densification, shrinkage, strength, hardness, necks, swelling and sintering atmosphere, to name a few [16, 18]. The authors in [19] show that oxide reduction is obtained at 0.5 - 0.8·Tmelt, around 500 and 800°C for Cu by isothermal sintering and porosity is still considerable for long sintering times. Also, they analyze the creep mechanism for Cu at 700-900°C and the considerable shrinkage after Cu is heated at a high rate over 500°C. The authors [20] discuss the relations between sintering temperature, time and pressure and the impact on density and strength for sintering temperature variations from 600 to 900°C. One of the biggest problems that occur late in the sintering cycle is swelling, when dissolved oxygen reacts with the sintering atmosphere to produce water vapour [21].

Another important issue deals with sintering atmosphere and vacuum. The authors in [4] discuss the effect of insoluble gases in such environments. For preventing oxidation and other effects, both Argon and Nitrogen are analyzed in [3]. Argon is recommended both in [3] and in [4], the latter dealing with copper graphite composites. Electronic systems are a massive opportunity for sintering and the innovative rapid sintering processes include a variety of heating approaches, including solar, microwave, induction, pulsed intense light, laser, magnetic pulse, capacitance discharge, spark plasma which are discussed in [16].

2.2 Spark plasma and microwave sintering

Spark plasma sintering (SPS) enables a powder compact to be densified by Joule heating when the pulsed direct current goes through the sintering specimen. This low voltage, pulsed direct current of high intensity and uniaxial pressure offers high heating rates (100-500°C/min) and short holding times (3-5 min.) in order to obtain fully densified bulk materials [22]. Authors in [16] underline the merits of SPS: rapid heating, short hold times, and possible electric field induced diffusion as current densities reach 1000 A/cm^2, where electromigration supplements diffusion. Authors in [3, 17, 23, 24] point out to the effects of densification and gain growth on nanosized particles, mainly the minimal grain growth under high densification rates. The authors in [22] observed that increasing SPS temperature led to greater hardness, elastic modulus and density for WC-Co cemented carbides. Authors in [17] associate the conductity increase with the relative density when sintering SiC. The authors in [24] observe the neck growth due to the current influence. Authors in [16] conclude that SPS delivers a higher sintered density at intermediate temperatures compared to conventional sintering, but at typical peak temperatures there is less advantage. In [15] the authors discuss the promising benefits of SPS for Cu-graphite composites in terms of hardness, electrical resistivity and volume density. Yet, when compared to conventional sintering, the cost is a major impediment [16].

Another method that rivals conventional sintering in terms of rapid heating rate is represented by microwave sintering. By absorbing microwaves, the metallic materials in powder form can produce nearly fully dense bodies [3]. Also, sintering under microwaves leads to a better homogeneity and to an improvement of the microstructure with a reduction of porosity and grain size. Other benefits include: relatively much shorter cycle time, finer microstructures leading to better quality products, substantial energy savings and eco-friendliness [3]. However, microwave sintering is a complex process, which is much more difficult to control than conventional sintering, especially in terms of uniform heating [4].

Promising results in terms of electrical resistivity are described in [7] when the copper powder was exposed to high electromagnetic field.

Also other sintering methods are of interest, like photonic sintering [23]. There are many studies regarding the sintering of copper and its composites. Authors in [16] have examined the electrical conductivity of copper after sintering and discussed how the particle size, density and temperature affect its surface, volume diffusion and grain boundary.

Results were already obtained at Transilvania University by microwave and plasma sintering of Cu-TiC-graphite (Fig. 2). Yet the reflection of microwave radiation due to the high content of cooper are major drawbacks in this particular case; so, the solar radiation is a promising solution for designing a green method for sintering cooper based metal powders.

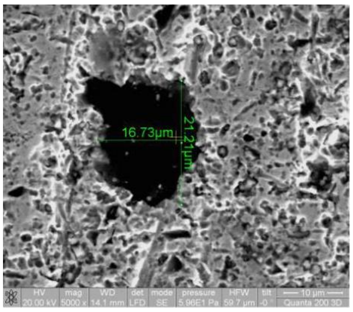

Figure 2. SEM microstructure of the Cu-TiC-graphite alloy

3. SOLAR SINTERING

3.1 Solar Energy Applications

Our recent research efforts are directed towards applying an unconventional sintering method based on solar energy for improving the mechanical and physical properties of Cu-TiC-graphite alloys while maintaining the electrical properties of copper. In the meantime we also ask the question if industrial sintering based on this method is economically feasible.

At present, industrial use of solar energy is primarily geared to converting light energy into electricity through the use of solar photovoltaic panels. At a relatively high level, solar energy is also used for desalination and purification of water, treatment of sludge and toxic waste, acceleration of biogas production and the production of hydrogen as an excellent non-polluting fuel. Such industrial technologies require temperatures up to 150 °C. For such processes, various solar heating systems using flat plate collectors or vacuum tubular collectors are successfully used [25].

Technological processes that use higher temperatures require concentrated solar power (CSP) equipment. These collectors can provide an extremely high temperature by using reflective panels that follow the sun and focus large amounts of sunlight on the heated object. Solar energy spreads almost radially and uniformly through space, but its spectral features do not change because it does not encounter matter with which to interfere. The energy flow decreases with the square of the distance from the sun. Thus, when the radiation reaches several hundred kilometers above the Earth's surface, the radiant flux is approximately 1360 W/m^2 [26].

Technologies using solar energy in the current production processes offer much greater potential for the future, as discussed in [27].

3.2 Solar Sintering Procedure

For solar sintering, existing research and testing equipment has been used at the PROMES-CNRS Solar Platform of Font-Romeu Odeillo - France research center . The technical means and research facilities in this institute offer the possibility of vacuum or atmospheric heating of samples to high temperatures, the heating and cooling rates being programmable. Also, the existing equipments allow analyzing the evolution in time of solar radiation that occurs during experiments, as well as for the evaluation of results [2].

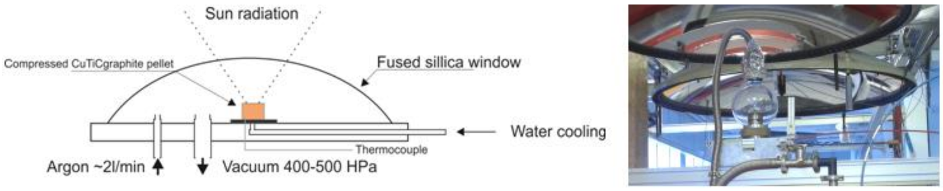

Figure 3. Proposed solution for sintering tests (left) and actual solution adapted to the solar concentrator operation (right)

After preliminary results at Transilvania University of Brasov, the optimized values of the powder ratio and milling time/ condition were determined as follows: Chemical composition: Cu 90%, TiC 5%, Graphite 5%.Our research was focused on the solar sintering of 20 different compacted pellets of Cu-TiC-graphite. The sintering was made in a solar furnace, as presented in Fig. 3.

3.3 Solar Sintering Cycles

The compacted pellets of Cu-TiC- graphite were sintered by heating and maintaining the pellets at a precise temperature in a controlled environment (Argon) at temperatures between 650 and 850°C for a maintain time of 20-30 min.

The other operational parameters range is: direct normal irradiation > 850 W/m^2, pressure ~ 400 hPa, Ar flow rate: 2l/min. The best, medium and worst radiant flux evolution in time per day is presented in Fig. 4, 5, and 6.

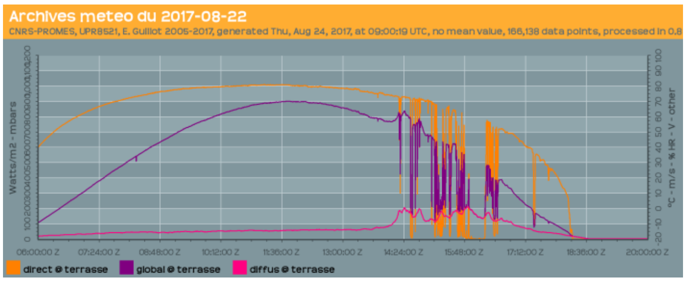

Figure 4. Best day radiant flux evolution

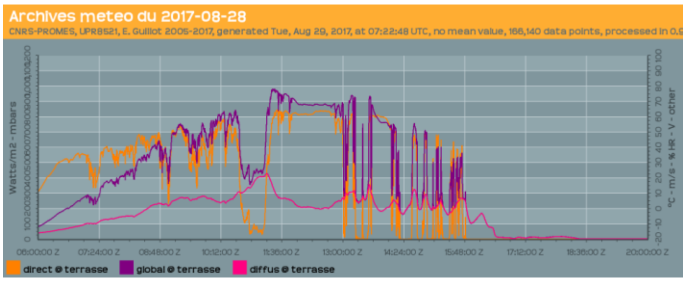

Figure 5. Medium quality day radiant flux evolution

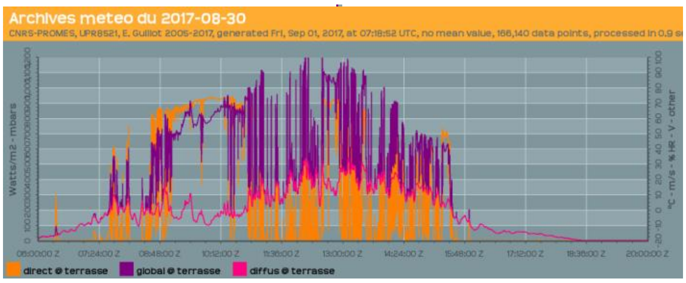

Figure 6. Worst day radiant flux evolution

A total of 6 days have proven to be useful for our experiments. Even under conditions of solar flux fluctuations, it was possible to obtain thermal stability for sintering in most cases, as can be seen in Fig. 7, 8 and 9. Under frequent fluctuations, both the number of sintered probes and sintering temperatures were reduced in order not to affect the probe integrity.

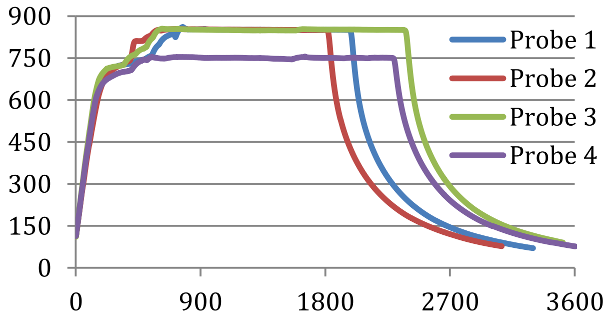

Figure 7. Temperature variation (°C) in time (sec.) for Fig.4

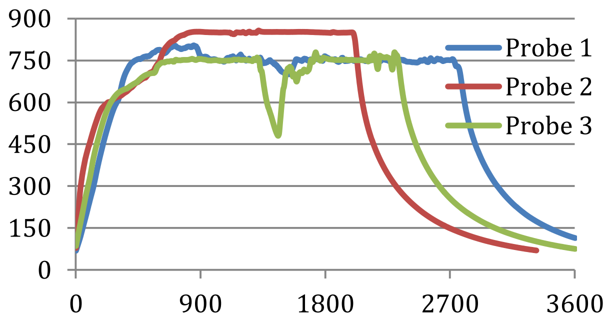

Figure 8. Temperature variation (°C) in time (sec.) for Fig.5

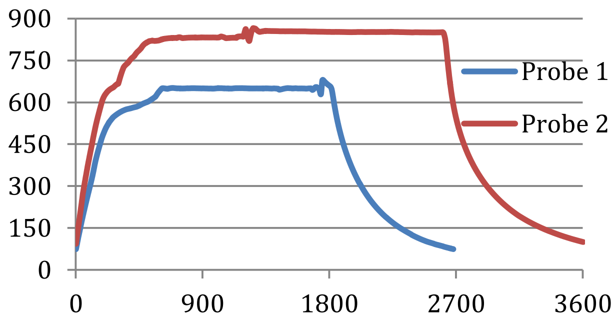

Figure 9. Temperature variation (°C) in time (sec.) for Fig.6

4. EXPERIMENTAL RESULTS

The mixture of the powder was made by mechanical mill using steel balls of 15mm diameter at a 600 rpm at a temperature of 40°C for 2 hours. The powder mixture was compacted at 510 MPa into a mold with the nominal diameter of 15mm and 15 mm high.

During the experimental tests, different temperature and sintering time values were tested in the vacuum environment, as can be seen in Table 1. Probe 11 (marked with red in Table 1) showed significant modifications in size in addition to swelling, which requires further investigation on the relations between compacting force and sintering setup and conditions, like sintering time and temperature.

While achieving thermal stability, as seen in Fig.7-9, and structural integrity of the probes, as seen in Table 1, electrical conductivity is another objective of the current study.

Table 1. Size modifications (initial diameter ϕ=16mm and height h=9mm) of 12 same composite probes after sintering, pressed between 100 and 150 kN, under different sintering cycles (MT=maintain time) without or with strong flux fluctuations (SFF)

Probe pressed at	Sintering conditions	Probe after sintering
1. 100kN	irad.=967-989 W/m^2, pres.=400-670hPa,T=850^{o}C,MT=20min.	ϕloss=0.21mm, h loss=0.33mm
2. 100kN	irad.=1005-1009 W/m^2, pres.=400-680hPa,T=850^{o}C,MT=20min.	ϕloss=0.23mm, h loss=0.21mm
3. 100kN	irad.=996-983 W/m^2, pres.=400-680hPa,T=850^{o}C, MT=30min.	ϕloss=0mm, h loss=0.22mm
4. 100kN	irad.=870-886 W/m^2, pres.=400-670hPa,T=850^{o}C, MT=25min.	ϕloss=0mm, h loss=0.07mm
5. 100kN	irad.=966-932 W/m^2, pres.=400-680hPa,T=750^{o}C, MT=30min.	ϕloss=0mm, h loss=0.12mm
6. 100kN	irad.=750-866 W/m^2, pres.=400-670hPa,T=750^{o}C, MT=20min.	ϕloss=0mm, h loss=0.14mm
7. 120kN	irad.=750-890 W/m^2, pres.=400-680hPa,T=750^{o}C, MT=20min.	ϕloss=0mm, h loss=0.05mm
8. 120kN	irad.=712-500 W/m^2, pres.=400-680hPa,T=750^{o}C, MT=30min, SFF	ϕloss=0.11mm, h gain=0.03mm
9. 120kN	irad.=750-830 W/m^2, pres.=400-680hPa,T=850^{o}C, MT=20min.	ϕloss=0.17mm, h gain=0.03mm
10. 120kN	irad.=830-600 W/m^2, pres.=400-680hPa,T=750^{o}C, MT=25min, SFF	ϕloss=0.05mm, hloss=0.07mm
11. 150kN	irad.=892-900 W/m^2, pres.=400-670hPa,T=850^{o}C, MT=20min.	ϕgain=0.31mm, h gain=0.55mm
12. 150kN	irad.=870-825 W/m^2, pres.=400-670hPa,T=750^{o}C, MT=20min.	ϕgain=0.33mm, hloss=0.31mm

Initial tests have revealed electrical resistivity values comparable to the ones obtained for the same composite microwave sintered at 850°C for 10 min.: $3.6 \cdot 10^{-8}$ to $15 \cdot 10^{-8}$ Ω·m.

Also, initial tests for probes 1, 2, 5 and 6 from Table 1 have revealed wear rates from $2 \cdot 10^{-4}$ to $5 \cdot 10^{-4}$ mm^3/Nm, while the friction coefficient varied from 0.3 to 0.6.

5. CONCLUDING REMARKS

The test cooper based pellets fabricated at Research Institute of Transilvania University of Brasov were sintered at the PROMES-CNRS facility in regard to the following objectives:

- Solar sintering of the Cu-TiC-graphite pellets by using different expose times, temperatures and a vacuum environment.

- Improvement of the mechanical proprieties of the solar sintered pellets compared to the already obtained plasma and microwave ones.

As presented in this paper, current research in the field of solar sintering of compacted powders proves to be a feasible method for obtaining high quality composite materials. The use of solar energy, besides the advantages of green energy, allows materials / powders to be sintered at much higher heating and/or cooling rates than conventional techniques, thus obtaining thermal stability.

By sintering the compressed powder pellets with the MSSF vertical furnace at the PROMES facility an optimized Cu-TiC-graphite alloy was obtained. Different powder ratios, compacting force and different solar heat sintering cycles used have led to a continuous layer (graphite) of the solid lubricant on the tribo-surface of the sintered alloy.

Initial tests at Transilvania Univeristy of Brasov have shown that electrical conductivity does not suffer a significant reduction while wear rate and friction coefficient are better or similar to those for pure copper, in accordance to the promising results obtained by microwave sintering and SPS. The following tests are under way: more mechanical tests (wear and micro-hardness), microscopy for structural modifications (SEM and EDS), more tests for determining electrical resistivity and conductivity, and thermal behavior tests (differential scanning calorimetric analysis).

The composite material of Cu-TiC-graphite sintered with solar energy has direct application in the fabrication of electrical contacts and antennas with the purpose to improve resistance, ductility and thermal stability without causing considerable decreases on electrical and thermal conductivity.

6. ACKNOWLEDGEMENT

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