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Microwave Ignited Combustion Synthesis as a Joining Technique for Dissimilar Materials

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Microwave energy has been exploited to ignite combustion synthesis (CS) reactions of properly designed powders mixtures, in order to rapidly reach the joining between different kinds of materials, including metals (Titanium and Inconel) and ceramics (SiC). Beside the great advantage offered by CS itself, i.e., rapid and highly localized heat generation, the microwaves selectivity in being absorbed by micrometric metallic powders and not by bulk metallic components represents a further intriguing aspect in advanced materials joining applications, namely the possibility to avoid the exposition to high temperatures of the entire substrates to be joined. Moreover, in case of microwaves absorbing substrates, the competitive microwaves absorption by both substrates and powdered joining material, leads to the possibility of adhesion, interdiffusion and chemical bonding enhancements. In this study, both experimental and numerical simulation results are used to highlight the great potentialities of microwave ignited CS in the joining of advanced materials.

Keywords intermetallics, joining, powder metallurgy

1. Introduction

Among the wide number of materials manufacturing procedures, combustion synthesis (CS) represents one of the most attractive approaches due to its several advantages with respect to the conventional ones. CS is based on the simply concept of taking advantage of the high exothermicity of several reactions, which become self-sustaining after a proper ignition step. Thus, CS is an energy saving procedure since the only external energy requirement is limited to the ignition step. The heat released by the exothermic reaction itself will provide the further energy required to reach a complete transformation into the desired products throughout the entire reactive specimen.

CS experiments can be conducted in two different ways depending on the way the ignition is reached: in the Self-propagating High-temperature Synthesis (SHS) mode of combustion the reaction is ignited at one end of the reactive sample and it self-propagates to the opposite side in the form of a combustion wave at very high velocities, while in the thermal explosion (TE) regime of combustion the whole sample is uniformly heated up to the ignition temperature so that the reaction takes place simultaneously throughout the volume (Ref 1-4).

In addition to the low energy requirements, the well recognized advantages of CS approach over more conventional techniques used to prepare advanced materials can be summarized as follows (Ref 5):

- the higher purity of the synthesized products, due to the high temperatures involved which can volatilize low boiling point impurities;
- the exothermic nature of the CS processes avoids the need of expensive and complex facilities and equipments;
- the fast kinetics typical of combustion waves result in low operating processing times;
- the high thermal gradients and rapid cooling rates can give rise to new non-equilibrium or metastable phases;
- materials can be synthesized and consolidated into a final product in one step using the chemical energy released by the interaction between the reactants.

Moreover, it has also been reported (Ref 6-9) that, when exploited to produce composite materials, CS offers a further important peculiarity, which is the excellent bonding between the reinforcement and the matrix components as a result of the in situ formation of such phases at high temperatures.

Such CS advantages can be proficiently exploited in advanced materials joining applications: due to the rapid and highly localized heat generation typical of CS processes, there is a limitation of thermal damage of heat-sensitive substrates, and hence the possibility to spatially limit the extension of heat-affected zones. Moreover, the great chemical compatibility between selected reaction products and the substrates must be highlighted, since the process used to produce the joint is often the same as the one used to produce the substrates.

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Joining between dissimilar materials, such as ceramic and metals, requires generally a smart design of a graded joining material to avoid abrupt changes from a substrate to the other. In this particular framework, it is well known that the combined use of powder metallurgy and CS techniques in functionally graded materials (FGMs) manufacturing can benefit from the fast kinetics involved, allowing to create non-equilibrium structures or to lead to products less prone to homogenization, thus preserving the gradient structure imparted during the forming step (Ref 10).

Among the wide number of ignition techniques, microwave (MW) energy possess selective and volumetric characteristics (Ref 11) (according to the dielectric and magnetic properties of the material to be treated) which can be proficiently exploited in the ignition and eventually in the sustaining of CS reactions for joining applications. Indeed, MW heating is based on energy transfer from the electromagnetic field directly into the joining material and subsequent heat generation due to polarization, conductive and magnetic losses.

The possibility of conveying energy into a material, according to its dielectric, electric, and magnetic properties can be summarized by the equation of the power density distribution (Eq 1), which describes the power dissipated per unit volume into the material by the conversion of the electromagnetic energy into heat (Ref 12):

$$P_{\mathrm{d}} = \omega\varepsilon_0\varepsilon''_{\mathrm{eff}}E^2_{\mathrm{rms}} + \omega\mu_0\mu''_{\mathrm{eff}}H^2_{\mathrm{rms}} \qquad \text{(Eq 1)}$$

where P_{d} = local value of the power density in the material (W/m^3); $\omega = 2\pi f$ (Hz), f = frequency of the incident MWs; $\varepsilon''_{\mathrm{eff}}$ = relative effective loss factor, including conductivity losses together with the polarization ones; μ''_{eff} = imaginary part of the relative effective magnetic permeability; E_{rms} = local electric field intensity (V/m); H_{rms} = local magnetic field intensity (A/m).

In order to quantify how deep (volumetric) the power from the electromagnetic field will penetrate into the material, until a given attenuation is achieved, the power penetration depth must be considered. For dielectric materials, a simplified equation (considering $\varepsilon'' \ll \varepsilon'$) describing the power penetration depth is given by Eq 2:

$$D_{\mathrm{p}} = \frac{\lambda_0\sqrt{\varepsilon'_{\mathrm{r}}}}{2\pi\varepsilon''_{\mathrm{r}}} \qquad \text{(Eq 2)}$$

where λ_0 is the wavelength of the incident MWs, $\varepsilon'_{\mathrm{r}}$ is the relative dielectric constant, and $\varepsilon''_{\mathrm{r}}$ is the relative loss factor (Ref 12).

On the other hand, in case of conductors, like metals, the conversion of the electromagnetic energy into heat occurs only in a very thin layer, whose thickness can be described by the skin depth, reported in the following Eq 3,

$$d = \sqrt{\frac{1}{\pi f\sigma\mu_0\mu'_{\mathrm{r}}}} \qquad \text{(Eq 3)}$$

where d = skin depth (m), σ = electrical conductivity (S/m).

Hence, bulk metals are considered reflecting materials at the MW frequencies. However, the situation changes in case of powdered metals.

At the typical ISM (allocated for Industrial, Scientific, and Medical applications) MWs frequency of 2.45 GHz, the skin depth value for most metals is of the order of micrometers, leading to the possibility of directly heat micrometric metallic powders by means of MW energy. However, MW interaction with powdered metals is still debated and the possible involved mechanisms could rely on induction of eddy currents due to the H field (Ref 13), on the presence of a thin dielectric oxide layer surrounding each metal particle (Ref 14) or on localized breakdown phenomena (Ref 15).

Macroscopically, as schematically reported in Fig. 1, the MWs absorption selectivity by reactive and micrometric metal powders mixture with respect to the reflecting behavior of bulk metallic substrates represents an intriguing approach to in situ synthesize the joining material by MWs ignited CS, leaving the substrates temperature almost unaltered during the heating process up to the ignition temperature.

In this case (reflecting substrates), the rapid heating rate typical of MW-assisted materials processing techniques, constitutes a further advantage in terms of time and energy saving as well as minimization of heat-affected zones.

MW absorption can occur also in the substrates, which then compete with the joining material in converting the electromagnetic field energy into heat, as shown in Fig. 2.

Sometimes, the products formed in the joining material can present significant MWs absorption. In this case, the electromagnetic energy source can be exploited in continuing to heat the joining region also after the completion of the CS reaction. This is expected to enhance chemical bonding and interdiffusion.

In all the cases, the highest temperature manifested during the whole process coincides with the combustion temperature reached in the joining material, thus the heat-affected regions of the joined components are usually very thin.

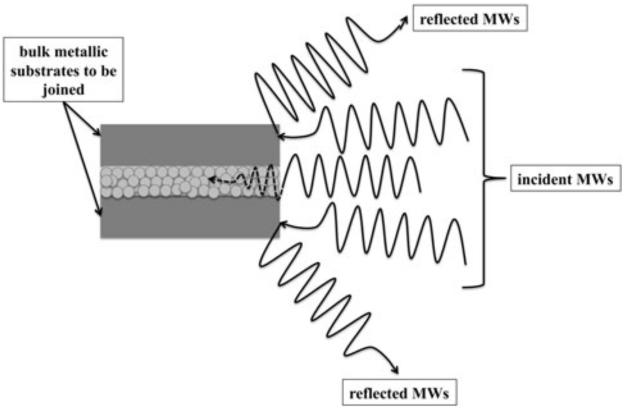

Fig. 1 Selective MWs absorption by compacted micrometric metallic powders positioned between two bulk metal reflecting substrates to be joined by MWs ignited CS reaction occurring between the powders

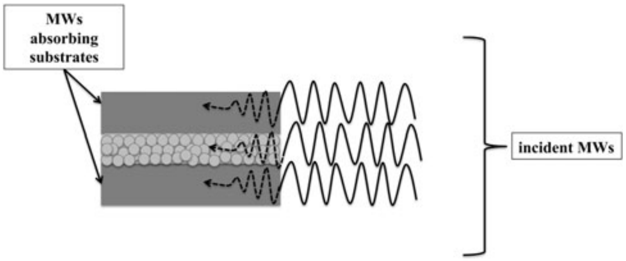

Fig. 2 Competitive MWs absorption by both micrometric metallic powders and not-reflecting substrates

Aim of the present work is to report results concerning the feasibility of the MWs ignited CS approach to join different kinds of materials (Titanium, Inconel and silicon carbide) using the CS of pressed Ni + Al or Si-C-Ti powders.

Particularly NiAl intermetallic phase was chosen as the joining material for Titanium substrates since it has already shown a great chemical compatibility, with formation of complex binary and ternary phases. The authors have already exploited CS of NiAl intermetallic phase, ignited by means of MW energy, in order to realize NiAl intermetallic coatings over grade 2 Ti substrates. The peculiar reaction conditions led to the formation of a thick coating with a duplex microstructure constituted of: (i) a polycrystalline outer layer essentially made of NiAl grains without preferential orientation; (ii) an intermediate Ni-Al-Ti layer with a mixed dendritic and eutectic microstructure (Ref 16, 17). This reaction interlayer greatly improved adhesion of the coating to the underlying substrate, demonstrating also to possess enhanced mechanical properties with respect to the outer NiAl coating (Ref 16) and good oxidation resistance up to 750 °C (Ref 18).

Ni-based superalloys possess also a great chemical compatibility with NiAl intermetallic phase due to their high Ni content (Ref 19-24), and this, together with the NiAl attractive combination of low density (5.9 g cm^{-3}), high melting point (1950 K), high strength, good corrosion and oxidation resistance, and high thermal conductivity (76 W m^{-1} K^{-1}) (Ref 20), led to the choice of such intermetallic phase as a promising material to realize sound joints between advanced metallic substrates.

On the other hand, as far as SiC-based components are concerned, they are very promising advanced ceramic materials in nuclear and high temperature power plants application fields (Ref 25). However, due to the harsh working conditions, very few elements are allowed to be used in joining materials for high temperature applications and, in particular, in a nuclear environment, since joints must be stable to transmutation reactions, show low activation after neutron irradiation and reliable chemical, thermal, and mechanical compatibility with silicon carbide (Ref 26). At this particular regard, Ti, Si, and C are considered suitable elements for joining materials in a nuclear environment (Ref 27) since they can react to give refractory phases, possessing the aforementioned requirements, through a CS approach ignited by MW energy (Ref 28, 29).

Multiphysics numerical simulation has been performed in case of both MWs reflecting and MWs absorbing substrates, in order to highlight the peculiarities of the here presented joining approach by MWs ignited CS.

2. Experimental

Elemental Ni (~3 μm, 99.7% purity), Al (-200 mesh, 99% purity), Si (-325 mesh, 99% purity), C (graphite syn, <20 μm), and Ti (-325 mesh, 99.5% purity) powders were all purchased from Sigma Aldrich (Milan, Italy).

Grade 2 Titanium (i.e., unalloyed commercially pure titanium) and Inconel, type 601 (with Ni ranging from 58 to 63 wt.%, Cr from 21 to 25 wt.% and Al from 1 to 1.7 wt.%) discs of 30 mm diameter and 1-2 mm thickness have been kindly provided by Akrapovic (Slovenia), while SiC discs have been purchased by Bettini Spa, Milan (Italy).

Powders were mixed in the desired stoichiometry, according to the CS reaction to be ignited.

Mixing was performed under vacuum in an Al_2O_3 ceramic jar for approximately 30 min, and then powders were uniaxially pressed (~150 MPa) to form thin reactive disc-shaped specimens of different weights and dimensions, depending on the extension of the areas to be joined.

CS experiments ignited by means of MW energy were all conducted in a TE_{10n} single mode applicator operating at the frequency of 2.45 GHz, which has been described in details elsewhere (Ref 16, 17, 30). Briefly, it consists of a magnetron generator, with an output power level ranging from 300 to 3000 W, connected to a three ports circulator and to a three-stubs tuner. The cavity for heat treatments consisted in a rectangular resonant applicator, based on the WR-340 waveguide (86 × 43 mm section). A shorting plunger allows controllably modifying the electromagnetic field distribution inside the cavity, thus having the maximum of the electric field in correspondence of the sample. A representation of the load disposition inside the single mode applicator is reported in Fig. 3, with the plunger removed. As clearly visible, the reactive compacted powders mixture is positioned between the two substrates to be joined, in order to realize a "sandwich-like" arrangement. According to this disposition inside the single mode cavity, the use of cylindrical refractory rams, perfectly fitting the cylindrical waveguide under cut-off conditions (visible in the upper side of Fig. 3) allowed applying a moderate pressure (approximately ranging from 0.15 to 1.5 MPa) during CS experiments, in order to enhance adhesion and reduce porosity in the newly combustion synthesized joining material, without any MW leakage. In order to avoid excessive oxidation a constant Ar flux (20 NmL/min) was blown into the single mode cavity during experiments.

β-NiAl joining material was obtained by CS according to the following exothermic reaction and was used in this work to join non-MW absorptive substrates (Ref 31):

$$\mathrm{Ni} + \mathrm{Al} \rightarrow \mathrm{NiAl} + 118\,\mathrm{kJ\,mol^{-1}} \qquad \text{(Eq 4)}$$

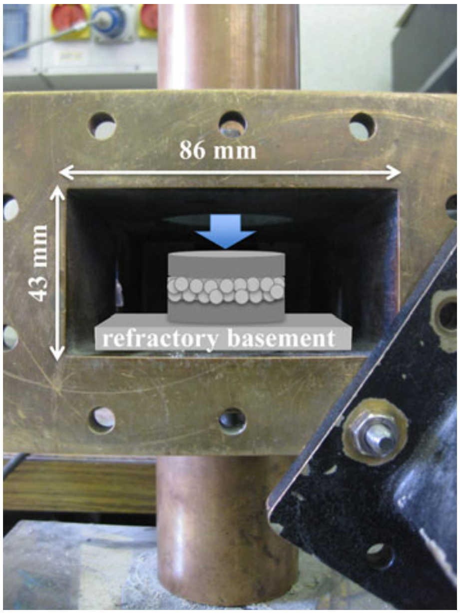

Fig. 3 Representation of the load disposition inside the single mode cavity. The arrow indicates the direction from which a moderate pressure is applied by cylindrical refractory rams (not depicted)

Formation mechanism of Nickel monoaluminide by a CS approach is known (Ref 32) to be governed by the dissolution of solid Ni into liquid Al, thus the ignition temperature (T_{ig}) of this process coincides with the Al melting point (~660 °C). Due to the high enthalpy value of NiAl phase formation, the adiabatic temperature (T_{ad}) of the NiAl CS greatly exceeds the melting point of the product (~1638 °C), reaching a value of approximately 1810 °C (Ref 32). The existence of liquid NiAl, even if for few seconds, favors reaction and interdiffusion with the metallic substrates (Ref 16), thus enhancing adhesion of the final joints.

In case of MW absorptive substrates, and in particular of SiC, the joining material was composed of Si-C-Ti powders (in a 1:1:1 molar ratio). In this ternary system, different exothermic reactions can be concurrently ignited leading to a complex mixture of reaction products, including different silicides and carbides, like TiC (T_{ig} in the 1300-1400 °C range (Ref 33, 34) and maximum combustion temperature ca. = 2600 °C (Ref 35)) and $TiSi_2$ (T_{ig} ranging from 600 °C to approximately 800 °C depending on the milling conditions applied (Ref 36) and maximum combustion temperature ranging approximately from 1130 to 1270 °C (Ref 37)), as demonstrated by the authors in previous works (Ref 38-40).

After CS completion and cooling under Ar flux, the obtained joined specimens were sectioned to investigate their microstructural and compositional features by means of scanning electron microscopy (ESEM Quanta-200 Fei, Oxford Instruments), energy dispersive X-ray spectroscopy (EDS, Inca-350, Oxford Instruments), and X-ray diffraction analysis (XRD, X'Pert PRO, PANAlytical, Almelo, The Netherlands) using Ni-filtered Cu Kα radiation (λ = 1.5405 Å).

2.1 Numerical Simulation Procedure

The authors have recently developed a simplified multiphysic model to numerically simulate the CS of intermetallic compounds ignited by MW heating (Ref 41), and to easily estimate the temperature distribution in all of the components involved in the joining process (Ref 40, 42). Briefly, the numerical simulation, described in detail elsewhere (Ref 40-42), was performed using the commercial software Comsol Multiphysics, coupling the reaction engineering module, the electromagnetic (RF) module and the thermal one. The simplified model developed assumed a temperature independent behavior of the reacting powders permittivity, the absence of any dimensional change as well as of any reaction between the reacting powders and the substrates to be joined, due to the small volume, and hence heat contribution, of the reaction layers experimentally measured.

The CS reaction occurring in the joining material was simulated inserting reaction kinetic parameters such as the Arrhenius pre-exponential factor and the activation energy as well as thermodynamic (entropy, enthalpy, and specific heat) and chemical-physical (concentration, density, and molecular weight) ones for all of the species involved in the reaction. The parameters used for the NiAl CS simulation can be found in (Ref 42) while the ones used for the Ti-Si-C joining system are reported in (Ref 40). The Reaction Engineering Laboratory module allowed studying the compositional and temperature variations in the joining material as the exothermic reactions proceeded.

The electromagnetic module was solved in the steady state, independently from the thermal module, due to the lack of reliable temperature-dependent data of materials parameters, for the model geometry reported in Fig. 4, which perfectly reproduces the experimental set-up used and described previously.

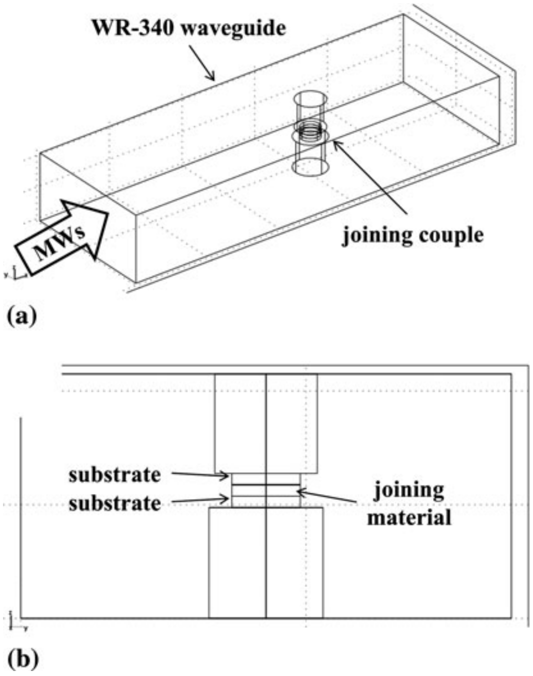

Fig. 4 Model geometry (a), reproducing the TE_{10n} microwave single mode applicator and the expanded front view of the joining couple disposition in the MW cavity (b)

Results obtained by the chemical and electromagnetic parts of the model, in terms of reaction heat and rate and MWs power density distribution, respectively, were then used as input parameters in the thermal module (which considers heat transfer by conduction and diffusion phenomena), allowing to estimate the temperature distribution before, during, and after CS occurrence, in each volumetric portion of the reactants and products as well as of the surrounding substrates and supporting materials.

3. Results and Discussion

3.1 Joining Between MWs Reflecting Substrates

The developed simplified model (described in section 2.1) allowed computing ignition (~616 °C) and maximum combustion (~1684 °C) temperatures in the Ni + Al joining material, and validating these data (which are in good agreement with literature results) with those obtained experimentally, as reported in previous works (Ref 31, 41).

Limitedly to the electromagnetic module of the developed model (Ref 40-42) solved for our experimentally realized joining couple arrangement, it was possible to highlight the selective MWs absorption by the micrometric metallic powders compact. In particular, Fig. 5 shows the electric field distribution along the applicator and the MWs power density distribution in the joining couple positioned in the center of the single mode cavity, at room temperature.

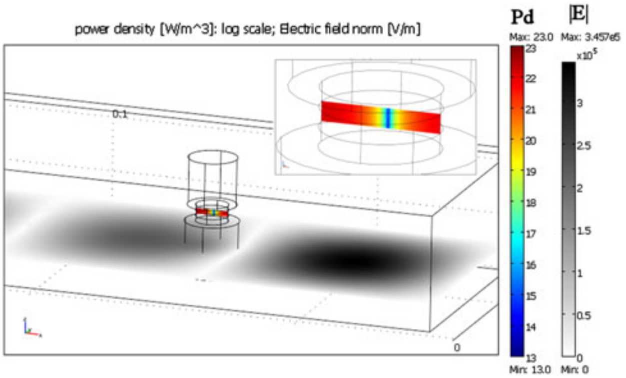

Fig. 5 Electric field (E) distribution (reported in gray scale) along the single mode applicator and MWs power density (P_d) distribution (reported in rainbow-like colored scale) in the joining couple, in the case of reflecting substrates. In the inset, an enlarged view of P_d distribution is reported

As reported in the zoomed view in Fig. 5, at the very beginning step of the joining process, the only MWs absorbing component in the whole load results the Ni + Al (50:50 at.%) compacted powders mixture, thus clearly confirming the selective nature of MWs heating technique in the particular application field of CS ignition.

MW-assisted CS of β-NiAl intermetallic phase has already proved to be an interesting route to form NiAl protective coatings on Ti, leading to a NiAl outer layer and an inner hypoeutectoid Ni-Al-Ti ternary layer (Ref 16-18).

CS of β-NiAl intermetallic (starting from a 50:50 at.% mixture of Ni and Al) phase has been ignited by means of MW energy in order to join Ti components and Ti to Inconel alloy (type 601).

According to the experimental procedure, in both cases 1.2 kW MWs output power was directed from the magnetron to the reactive specimen, which underwent ignition approximately in 2 min (considering also the time necessary to reach the proper impedance matching). A pressure of 0.15 MPa was applied during CS experiments.

XRD analysis was performed on the cross section of the joining material and reported in Fig. 6, which confirmed the obtainment of β-NiAl phase as the main product, with the presence of a small amount of Ni_3Al as secondary phase.

SEM images of the interfaces between the newly combustion synthesized β-NiAl phase and Inconel and Ti substrates are reported in Fig. 7(a) and (b), respectively.

For NiAl-Ti interface (Fig. 7b) the microstructure shows typically mixed dendritic and eutectic features, which originate from the reactions between liquid NiAl and the Ti substrate (Ref 16-18). Its thickness ranges approximately from 50 to 100 µm. The high exothermicity of the β-NiAl in situ formation allowed also its adhesion with the Inconel 601 substrate, as reported in Fig. 7(a). Aluminum diffuses from the NiAl phase toward the Ni-based superalloy substrate (Ref 43), as visible from the slightly darker middle layer (labeled in Fig. 7a) and from the EDS line scan of Al, superimposed to Fig. 7(a).

3.2 Joining Between MWs Absorbing Substrates

Silicon carbide (SiC)-based materials are known to be good MWs absorbers and they are often used in MW processing of materials as "susceptors" or co-absorbers in order to obtain more uniform temperature distributions and better electromagnetic field homogenization, decreasing at the same time electric arcs occurrence inside the MW cavity (Ref 30, 44-46).

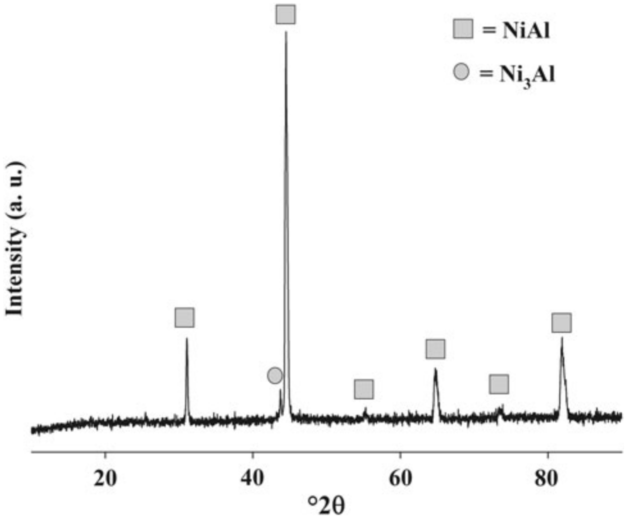

Fig. 6 XRD pattern of the combustion synthesized β-NiAl based joining material used for MWs reflecting substrates

By using the same simplified model mentioned in section 2.1, it was possible to obtain the MWs electric field and power density distribution in the case of joining between SiC-based components (Fig. 8), as well as to estimate the combustion parameters as detailed elsewhere (Ref 40). From the inset in Fig. 8, it clearly appears that in case of MWs ignited CS joining of SiC-based components, MWs are absorbed both by the reactive powders and by the substrates, but to a different extent. In particular, due to the different room temperature dielectric properties of the substrates and of the powders mixture, modeling results show that the power density, and hence heat generation, is much higher in the two outer SiC discs at room temperature.

As heating proceeds, the Si-C-Ti powders mixture is ignited but exposed to a much hotter environment with respect to the case of MW reflective substrates. Moreover, the CS products of the Si-C-Ti system are still good MW absorbers, and heat generations continues in the joining region, even after CS, until MW power is switched off (Ref 31).

This is expected to enhance adhesion and possible reactions between the substrates and the joining material. Interfacial microstructure observation confirms this expectation, as shown in Fig. 9, in which the porous SiC substrate exhibits good adhesion with the CS obtained joining material in the ternary Si-C-Ti system. The main phases constituting the joining material after CS resulted $TiSi_2$ and TiC as detailed elsewhere (Ref 38-40).

4. Conclusions

MW energy has been used in the present work to ignite CS reactions occurring in the joining material positioned between different kinds of materials. The here proposed approach has been demonstrated to benefit from both the highly localized heat generation (typical of CS approach itself) and the MW absorbing selectivity by micrometric metallic particles compacts with respect to bulk reflecting metal substrates. Good

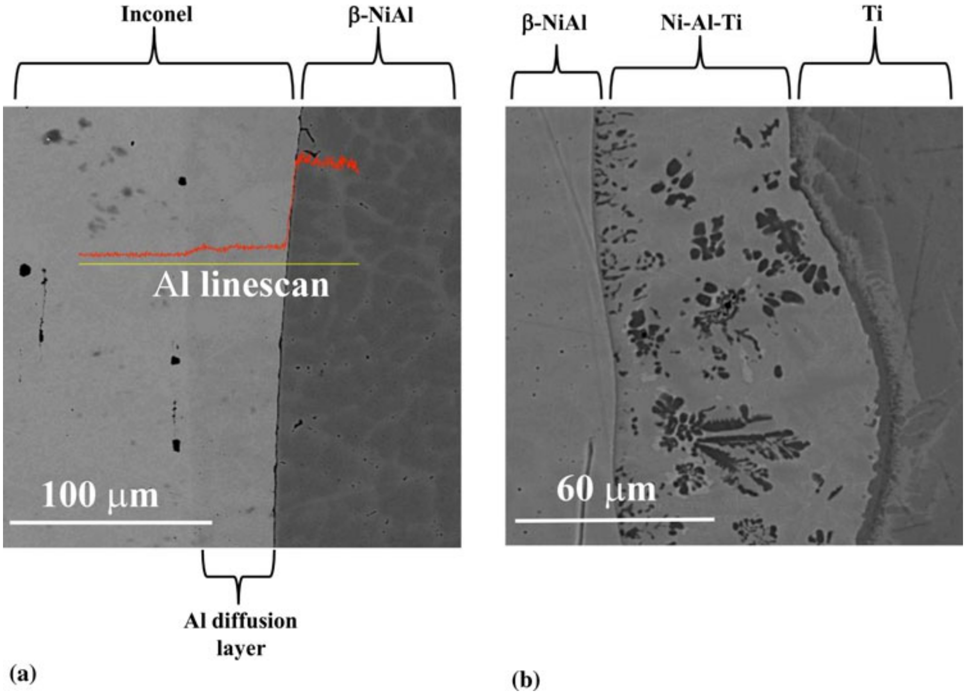

Fig. 7 (a) Interface between CSed β-NiAl and Inconel substrate; EDS Al line scan across the cross section shows Al diffusion from the intermetallic phase toward the Ni-based superalloy substrate. (b) Ni-Al-Ti reaction layer formed after joining at the interface between titanium component and MWs combustion synthesized β-NiAl phase

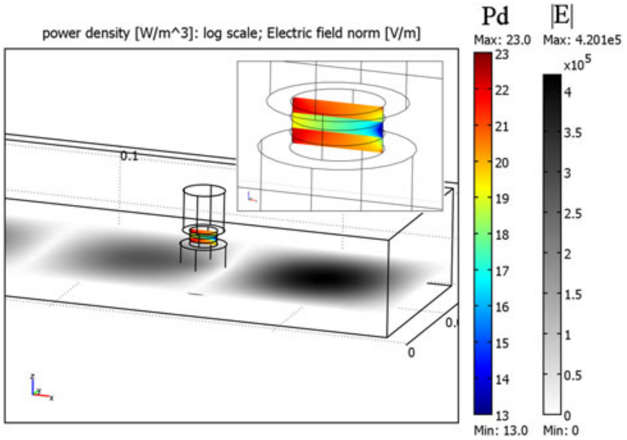

Fig. 8 Electric field (E) distribution (reported in gray scale) along the single mode applicator and MWs power density (P_d) distribution (reported in rainbow-like colored scale) in the joining couple, in the case of MWs absorbing substrates. In the inset, an enlarged view of P_d distribution is reported

joints were obtained between titanium and Inconel 601 alloy, by igniting the CS of β-NiAl intermetallic phase starting from a 50:50 at.% of Ni and Al powders, due to the formation of a ternary Ni-Al-Ti reaction layer and to the Al diffusion from the combustion synthesized aluminide to the Ni-based superalloy.

When MWs ignited CS technique was exploited to join MWs absorbing components, like SiC-based materials, it has been shown the competition in MWs absorption between the substrates and the reactive powders. Although this hides the selective nature of dielectric heating, it can enhance conversion degree of the starting reactants, adhesion, interdiffusion, and chemical bonding between the CSed products and the substrates.

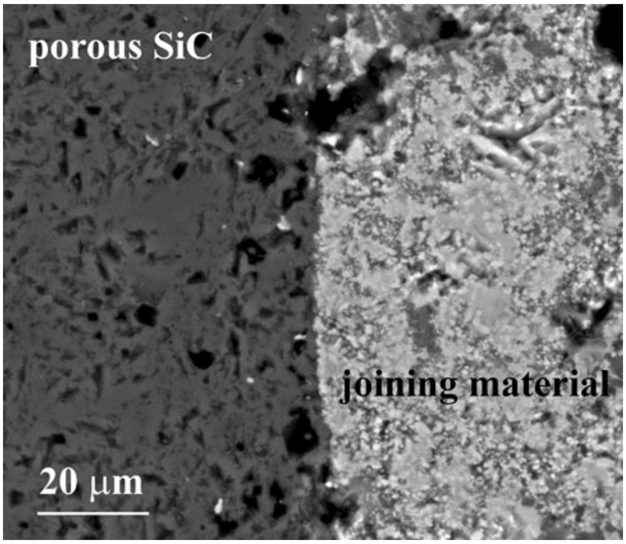

Fig. 9 SEM micrograph of the interface between porous SiC substrate and the products obtained by MWs ignited CS of Si-C-Ti powders mixture

These two aspects can be proficiently combined in realizing mixed joints between MWs reflecting (like metals) and MWs absorbing (some ceramics or composites) components.

Further research activity is currently in progress toward this direction, investigating also the possibility to use opportunely tailored functionally graded joining materials to be ignited.

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