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Microwave processing of sprayed alumina composite for enhanced performance

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Abstract

To meet the challenging in-service requirements, surfaces of many mechanical components need to be modified. This can be achieved by altering the surface properties through deposition technique followed by suitable post processing. Apart from the conventional methods, laser treatment is the most popular post processing technique. Of late, microwave heating is emerging as one of the potential post processing sources. Microwave processing of materials is fundamentally different from traditional techniques. In microwave processing, energy is directly transferred to the material through interaction of electromagnetic waves with molecules leading to volumetric heating. However, for processing of transparent (to microwaves) materials, a technique known as microwave hybrid heating (MHH) can be used. In this study, MHH is used for post processing of plasma deposited alumina–titania ceramic composite coatings on steel substrate; resulting properties of the post processed coatings are evaluated. Results show reduction in porosity, enhancement in microhardness, and wear resistance of the irradiated coatings owing to microstructural changes and densification. Results are discussed with suitable illustrations. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Microwave processing; X-ray methods; Porosity; Wear resistance; Acoustic emission; Al_2O_3; Plasma spraying

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1. Introduction

Performance of most of the mechanical components depends critically on their surface properties. Surfaces are subjected to different types of loading including mechanical, thermal, chemical, radioactive, etc., which can be severe with the increase in complexity of the working environment. This leads to continued degradation of the exposed surface. However, by suitable modification of the chemistry and metallurgy of the surfaces, it is possible to enhance the surface properties and hence the life of a component. This can be achieved by depositing a suitable chemically passive material (single or composite) onto the candidate surface. Most of the ceramics/ceramic composites, owing to their chemical stability, low density, hot hardness and wear resistance are preferred to their metallic counter parts as spray deposit material. Among the spraying processes, thermal spray deposition techniques have gained worldwide acceptance as the most useful method for depositing a wide variety of materials including ceramics and ceramic composites, which are difficult to process otherwise.[1,2] Thermal spray deposited surfaces, on the other hand, exhibit higher order roughness, inferior bonding quality[3] and generally contain voids such as (1) pore between flattened particles and (2) vertical cracks arising from thermal shrinkage of particles during and/or after spray processing.[4] However, thermally sprayed coatings' performance can be reasonably improved via different post treatment.[5,6] Published literatures in the field indicate there are three groups of post treatments:[6]

- Thermal processes (example: laser melting, hot isostatic pressing)
- Mechanical processes (examples: rolling, squeezing, grinding, polishing, lapping)
- Sealing process.

In the present study, post processing of thermally sprayed alumina (Al_2O_3)-titania (TiO_2) ceramic composite coatings has been achieved through an entirely different approach, and can be termed as 'Irradiation

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Process'. In this process, also termed as 'Microwave Glazing', thermally deposited ceramic composites were irradiated using high frequency microwave (Mw) radiation for a specified duration. The following sections discuss the basics, experimental procedure adopted and the consequent results of the process.

2. Microwave irradiation process

Microwaves are electromagnetic waves that have a frequency range of 0.3–300 GHz and corresponding wavelengths ranging from 1 m to 1 mm. Like lasers, they are highly coherent and polarized. However, unlike laser heating, microwave heating is fundamentally different from conventional heating processes. In conventional thermal processing, energy is transferred to the material through conduction, convection and radiation of heat from the surface of the material. On the other hand, microwave energy is delivered directly to materials through molecular interaction with the electromagnetic field. In heat transfer, energy is transferred due to thermal gradients, but microwave heating is the transformation of electromagnetic energy to thermal energy and is energy conversion, rather than heat transfer. The molecular structure affects the ability of the microwave to interact with materials and transfer energy.[7] As microwave radiation penetrates and interacts with molecules; transfer of electromagnetic energy takes place throughout the volume of the material, leading to volumetric heating. This, unlike conventional processes, ensures rapid and uniform heating of the material. As a result, the thermal gradient and the flow of heat in microwave processed materials are the reverse of those in materials processed by conventional heating. The degree of interaction (or absorption) of microwaves by a dielectric material is related to the material's complex permitivity, ε^* (F/m), and is defined as,[8]

$$\varepsilon^* = \varepsilon' - j\varepsilon'' = \varepsilon_0\left(\varepsilon_r - j\varepsilon''_{\text{eff}}\right) \tag{1}$$

where,

ε' dielectric constant
ε'' dielectric loss factor
ε_0 $= 8.86\times10^{-12}$ F/m, permitivity of free space
ε'_r relative dielectric constant, a measure of the polarisability of a material
$\varepsilon''_{\text{eff}}$ effective relative dielectric loss factor
j $= \sqrt{-1}$

During irradiation, microwaves penetrate and propagate through a dielectric material that generates internal electric fields within the bulk of the material; the electric fields induce translational motions of free or bound charges and rotate charge complexes as dipoles. The ability of dipoles to respond to the oscillatory electric field and their subsequent inability to keep up with rapid reversals in the field result in dielectric losses that are manifested as heat. The mechanism by which electromagnetic radiation interacts with materials in the microwave frequency range is yet to be understood well.[8,9] Since several mechanisms, including electrical conductivity, can operate simultaneously, for convenience, the loss mechanisms are well combined together in one loss parameter, and is expressed as,[9]

$$\varepsilon''_{\text{eff}} = \varepsilon'_r \tan\delta \tag{2}$$

The power absorbed, P (W/m^3) per unit volume, that provides the basis for heating is given by,

$$P = \sigma|E|^2 = 2\pi f\varepsilon_0\varepsilon'_r\tan\delta|E|^2 \tag{3}$$

where,

E is the magnitude of internal field (V/m).

It is also important to note that the depth of penetration of microwave through a given material varies appreciably with frequency. While highly conducting metals are opaque (zero penetration), low loss insulators like Al_2O_3, TiO_2 and ZrO_2 are transparent to microwaves at ambient temperature. This is due to the fact that, the electric fields are attenuated as the microwaves penetrates and propagates through the bulk material depending on their loss modulus, tan δ.

From the above discussion, it is clear that the relative dielectric constant (ε_r') and the loss modulus (tan δ) are the two parameters that describe the behaviour of a dielectric under the influence of a microwave field.

During heating, both ε_r' and tan δ change with temperature. For many ceramics, including Al_2O_3 TiO_2 and ZrO_2, tan δ is low at ambient temperature, but increases significantly as the temperature of the material increases. Beyond a certain material dependant critical temperature T_c, the rate of increase in $\varepsilon''_{\text{eff}}$ changes significantly.[9] For processing of such materials, a useful technique known as 'microwave hybrid heating' (MHH) is used effectively to raise the processing temperature beyond the threshold temperature (T_c) by placing the transparent/partially transparent material inside a highly absorbing material like SiC/charcoal. The observed temperature profile during post processing through MHH is illustrated in Fig. 1. It is seen that there is a sharp rise in temperature of the alumina-titania ceramic composite after around 1 min of microwave irradiation. During the initial period, the activated charcoal absorbs microwave and raises the temperature above T_c facilitating effective coupling of microwave with the ceramics and hence processing of the spray deposits. The two observed peaks P_1 and P_2 in the

Table 1
Details of materials

	Materials	Particle size (micron)	Thickness (mm)
Substrate	Low carbon steel	–	5
Bond coating	Nickel–aluminum (Ni–Al)	40–50	0.1
Top coating	Alumina (Al_2O_3) + 13 wt.% titania (TiO_2)	40–50	0.9

profile correspond the two phases alumina and titania present in the composite system.

Although limited published literatures are available[7–13] regarding processing of ceramics through microwave, most of them are concerned with sintering of ceramics. However, in this investigation, post processing of plasma spray deposited alumina-titania composite material has been carried out successfully by employing MHH technique. Post treated ceramic composite properties are evaluated, and preliminary results are discussed in this paper with suitable illustrations.

3. Experimental procedure

The methodology adopted and experimental details are presented in the following sections.

3.1. Materials and substrate preparation

Among the structural oxidic ceramics, alumina is widely used. Addition of dispersion, such as titania reduces the inherent brittleness of alumina and improves its tribological characteristics. In this study, alumina + 13 wt.% titania (AT-13) ceramic composite was used for spray deposition on low carbon steel substrate of dimension 60×10×5 mm. Substrates were cleaned and degreased in acetone medium. Prior to deposition, substrates were shot blasted to create mechanical roughening to facilitate mechanical keying/bonding; and also to induce compressive stresses on the surface to be coated. Metallic nickel–aluminum (Ni–Al) powder was used as bond coating material. Material details are presented in Table 1.

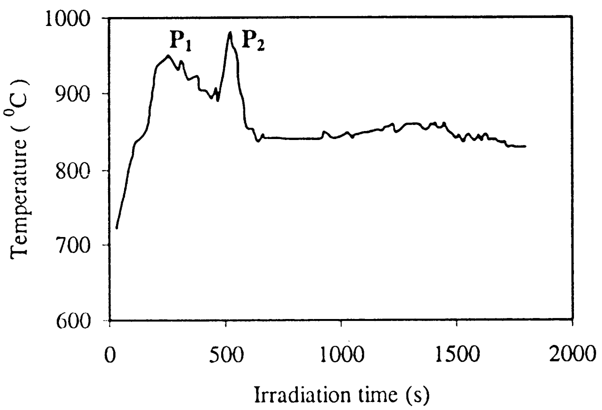


Fig. 1. Typical observed temperature profile during microwave irradiation.

3.2. Spray deposition

Spray powders were preheated to remove moisture content, if any, and to ensure free flowing. Preheated AT-13 powder was plasma deposited through atmospheric plasma spraying facility in the ambient condition on the preheated substrates maintaining a total spray deposit thickness of 0.9 mm. A nickel–aluminum interlayer of 0.1 mm was deposited prior to ceramic composite deposition. The spraying parameters are presented in Table 2. Spray deposits were diamond ground to attain uniform coating thickness and surface texture.

3.3. Post treatment

Post processing of the spray deposits was carried out in a domestic 1 kW multi-mode cavity microwave oven. Ceramic composite coatings were exposed to microwave radiation of 2.45 GHz for 30 min. Alumina–titania being transparent to microwaves at low temperature, the spray deposits were placed in relatively higher absorber activated charcoal environment to facilitate hybrid heating. Metallic substrates were selectively masked so as to avoid microwaves getting reflected. During irradiation, initially, charcoal couples with microwave and temperature increases. Once the temperature increases beyond the critical temperatures of the ceramics, they start coupling with microwaves and temperature rises rapidly as can be seen by the two distinct peaks in Fig. 1. Temperature during the irradiation was measured by an ULTIMAX IR pyrometer at a distance 1m from the source. Charcoal layer of the top surface of the ceramic coatings could be focused. Thus, the temperature profile in Fig. 1 represents the trend of

Table 2
Plasma spraying parameters

Parameters	Description
Current	800 A
Voltage	32 V
Power	40 kW
Arc gas pressure	0.4 MPa
Powder flow rate	60 gm/min
Spray stand off	130 mm
Gun traverse speed	18 mm/s

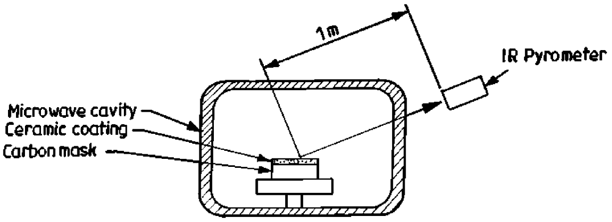


Fig. 2. Schematic of post processing and temperature measurement during MHH.

variation of the post processing temperature. A schematic of the irradiation and temperature measurement system is illustrated in Fig. 2.

3.4. Property evaluation

Microhardness was measured using standard microhardness testing facility. Porosity was determined through the optical method from scanning electron micrographs at random locations of the deposits. An average of three samples were taken. To evaluate the sliding wear resistance, deposits were tested through standard pin-on-disc arrangement at different normal loads in ambient condition. A hardened steel disc of hardness 65RC was used as counter surface.

Erosion wear resistance of the spray deposits was evaluated by subjecting them to high frequency impulse in an ultra sonic frequency generating facility using boron carbide (B_2C) as abrasive in ambient condition. Off-line weight measurement was carried out in a microbalance with 0.01 mg resolution after cleaning the specimen with acetone medium and subsequent drying. Acoustic emission signals emanated during wear tests were acquired through AET 5500 data acquisition system (DAS). Details of the wear tests are presented in Table 3. The schematic of the sliding and erosion wear test set ups are illustrated in Figs. 3 and 4 respectively.

4. Results and discussion

Alumina–titania ceramic composite coatings on low carbon steel substrate have been developed and ground with diamond. To investigate possible improvements in their properties, the diamond ground AT-13 composites were post processed (glazed) through microwave irradiation. The properties of both as-sprayed and irradiated spray deposits were evaluated through different tests. Results are discussed in the following sections.

Table 3
Details of wear tests

Sliding wear test		Erosion wear test	
Test	Pin-on-disc	Frequency	20 kHz
Counter face	Hardened steel (65RC)	Amplitude	50 micron
Normal load	10 N, 15 N and 20 N	Static load	5 N, 10 N and 15 N
Sliding velocity	5 m/s	Abrasive	Boron carbide
Temperature	Room temperature	Abrasive size	280 grit
Medium	Air	Abrasive:water	1 : 4
		Angle of impact	90°

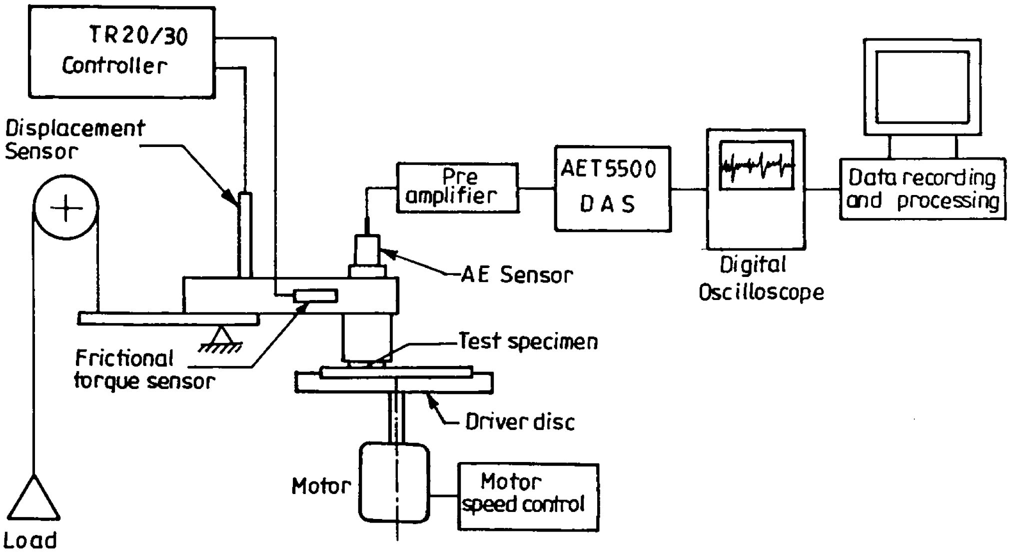


Fig. 3. Schematic of sliding wear test set up.

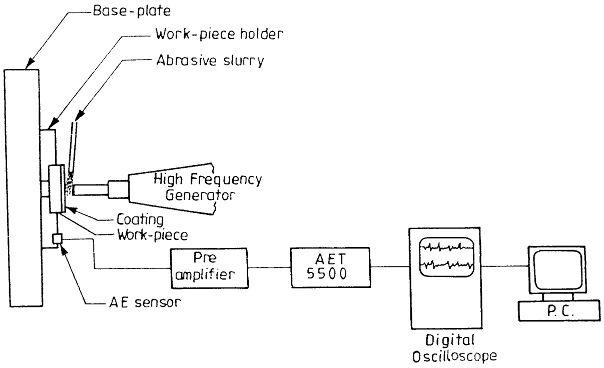


Fig. 4. Schematic of erosion wear test set up.

4.1. Phase analysis through X-ray diffraction (XRD)

The XRD profiles of the as-sprayed as well as post-processed spray deposited ceramic composite coatings are illustrated in Fig. 5. It is observed that, most of the stable and harder α-alumina in the powder phase is transformed to metastable tougher γ-alumina facilitating material flow/spreading. This is because of rapid cooling of the molten/semi-molten alumina powder particles immediately after deposition. Further transformation of α-alumina to γ-alumina is observed during post processing. Thus, the post-processed ceramic composite is mostly dominated by metastable γ-alumina phase. The presence of high temperature β-Al_2TiO_5 phase at room temperature in the post processed ceramic composite is attributed to the possible alumina-titania interaction/diffusion during irradiation. The highly suppressed intensity peaks and marginal shifts in the (intensity) peaks observed in the post-processed ceramic composite is attributable to the lattice straining due to densification during microwave irradiation.

4.2. Study of microstructure

A schematic of the cross section of spray deposits is illustrated in Fig. 6. The entire coating thickness has

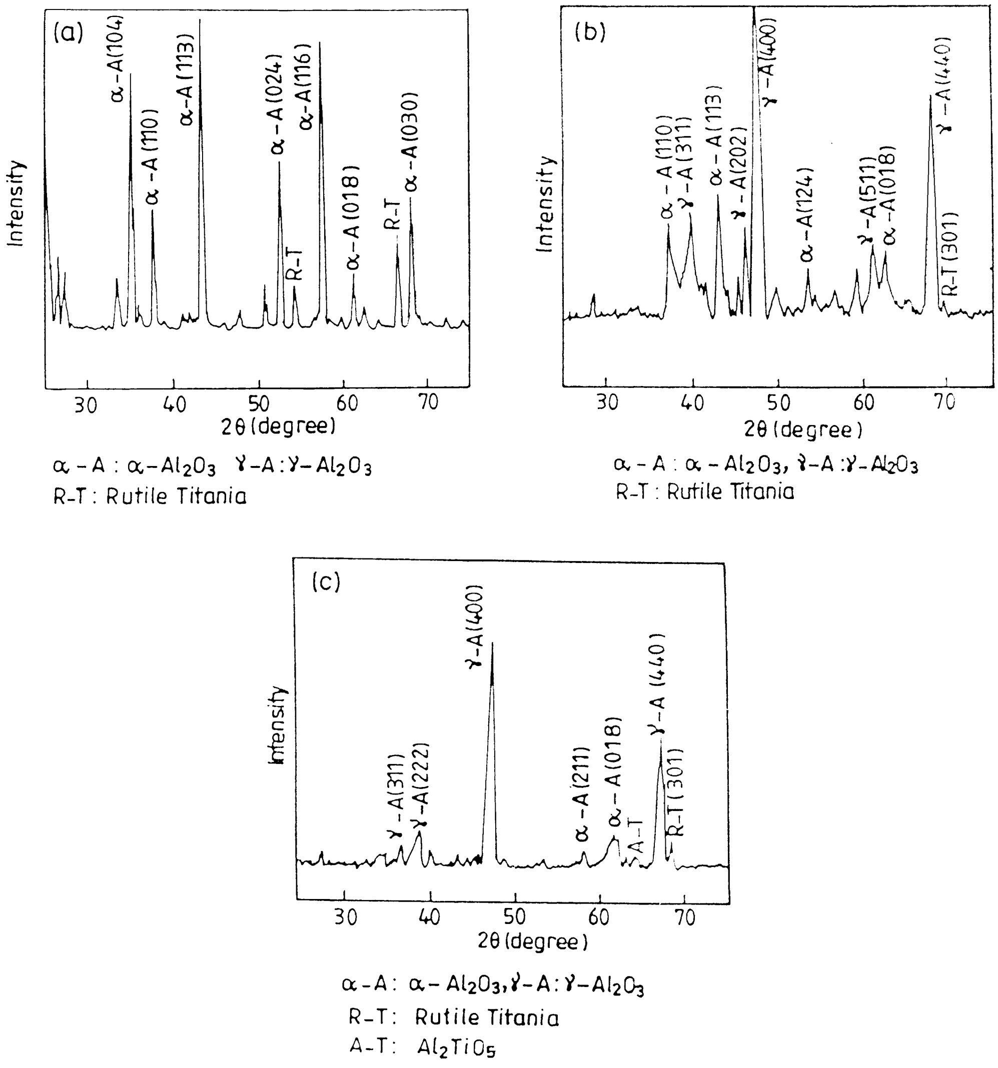


Fig. 5. Typical observed XRD profile of (a) spray powder, (b) as-sprayed deposit, and (c) microwave treated alumina-titania spray deposits.

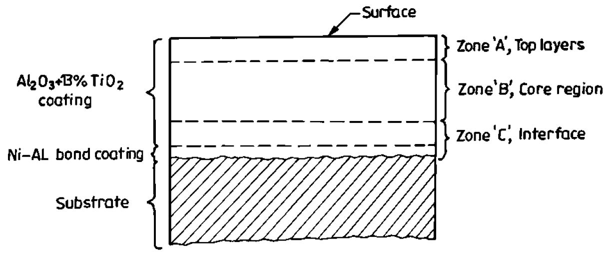


Fig. 6. Schematic of the cross section of spray deposits.

been logically divided into three different zones—'A', 'B' and 'C' for the purpose of analysis as shown in the Fig. 6. All the three zones, including the surface of the AT-13 spray deposits, both as-sprayed and post processed, were examined under the scanning electron microscope (SEM). Micrographs, typical of the surface as well as that of the three zones are presented in Figs. 7–10. While relatively coarse splats are clearly seen on the top surface of the as-sprayed deposits, a glazed texture can be seen on the irradiated surfaces as illustrated in Fig. 7. This is attributed to the possible flow of ductile γ-alumina phase during prolonged exposure to microwave. During MHH, as soon as the temperature crosses the threshold/critical temperature (T_c) of the alumina–titania system owing to microwave absorption by surrounding charcoal, at around 60 s of exposure, the AT-13 starts absorbing microwave and the temperature within the bulk rises sharply due to alumina and titania as seen in Fig. 1. This sets up a reverse gradient of heating, i.e. the core of the ceramic composite becomes hotter than the surface. While the heat transfer from the surface to the surrounding is more, the core temperature is retained for reasonably longer time leading to grain growth. This favours the observed epitaxial grain growth as can be seen clearly (Core region, zone 'B') in Fig. 8 (b). The usual layered structure of the plasma deposits is observed to be disturbed resulting densification of the spray deposits. The core section of the as-sprayed deposits (Fig. 8(a)), on the other hand, depicts pores as well as loosely held sprayed particles. However, owing to relatively better heat transfer through the surfaces, the top layers (zone 'A') of the spray deposits are not much distorted as illustrated in Fig. 9. The structure clearly indicates flow of material (γ-Al_2O_3) in the irradiated sample as seen in Fig. 9(b).

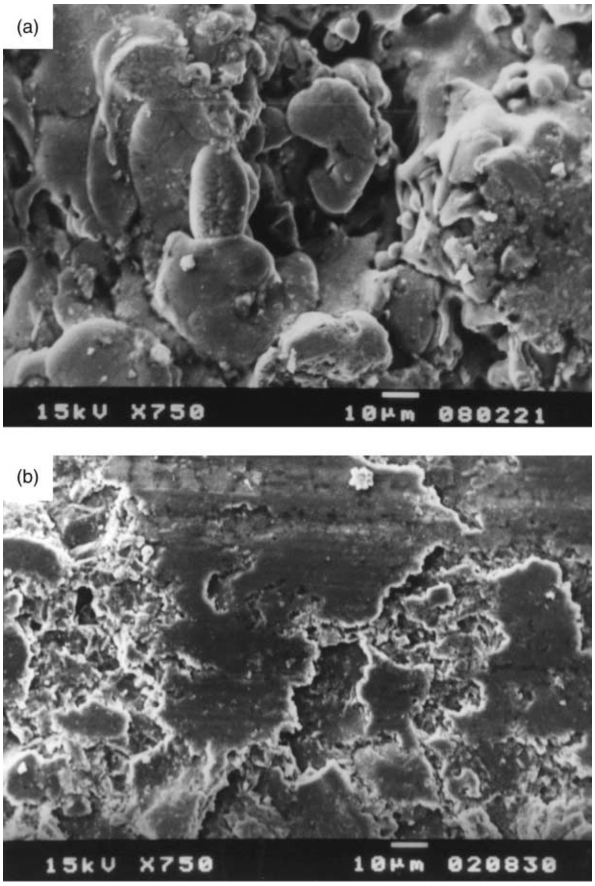


Fig. 7. Typical SEM micrographs of (a) as-sprayed surface showing coarse splats, and (b) post treated spray deposits with glazed texture.

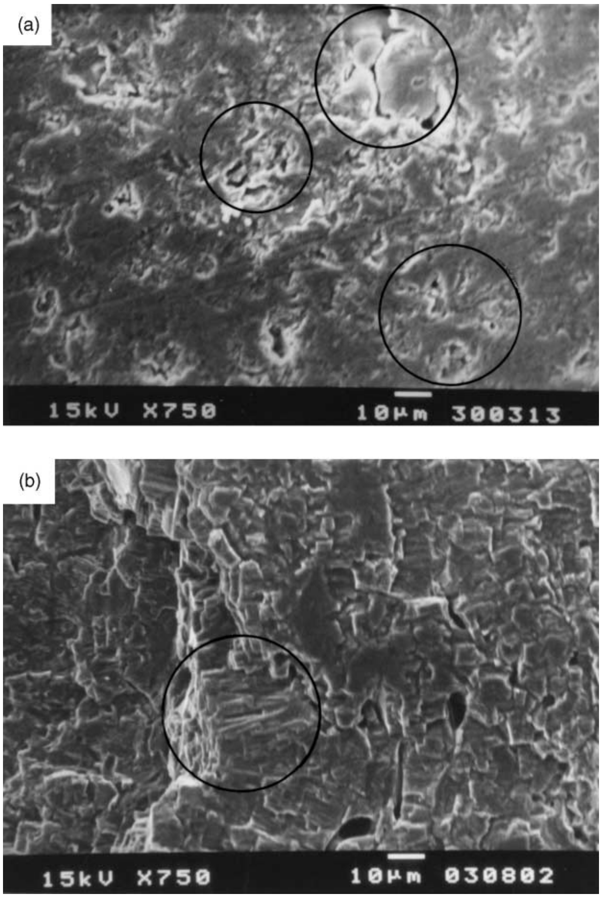


Fig. 8. Typical SEM micrographs of the Core region (Zone 'B') of (a) as-sprayed deposits that shows loosely held particles and pores in the marked areas, and (b) microwave treated spray deposits illustrating disturbed structure.

4.3. The interfacial structure

Typical micrographs of the coating-substrate interface (zone 'C') of the as-sprayed and microwave treated ceramic composites are illustrated in Fig. 10. Presence of metallic Ni–Al bond coating at the interface facilitates higher order of heat transfer through the substrate. While the as-sprayed coatings exhibit presence of voids, only marginal heating effect is seen in terms of remelting and resolidification in post processed ceramic composite material.

4.4. Porosity

Atmospheric plasma deposited ceramic composite coatings are inherently porous. While porosity could be beneficial in certain aspects, it reduces strength properties. Higher level of porosity also decreases tribological performance of coatings by increased tendency to spalling/ dislodgement. Heat treatment/post processing significantly influences the porosity level in the spray deposits. Typical results are illustrated in Fig. 11. As reported earlier, microwave irradiation induces $\alpha \rightarrow \gamma$-alumina phase transformation owing to rapid cooling. The $\alpha \rightarrow \gamma$-$Al_2O_3$ phase transformation is associated with an increase in volume leading to the observed densification, hence reduction in the percent porosity of spray deposit. Further transformation to stable and harder α-Al_2O_3 with a possibility of increase in porosity is most unlikely during decoupling of microwave

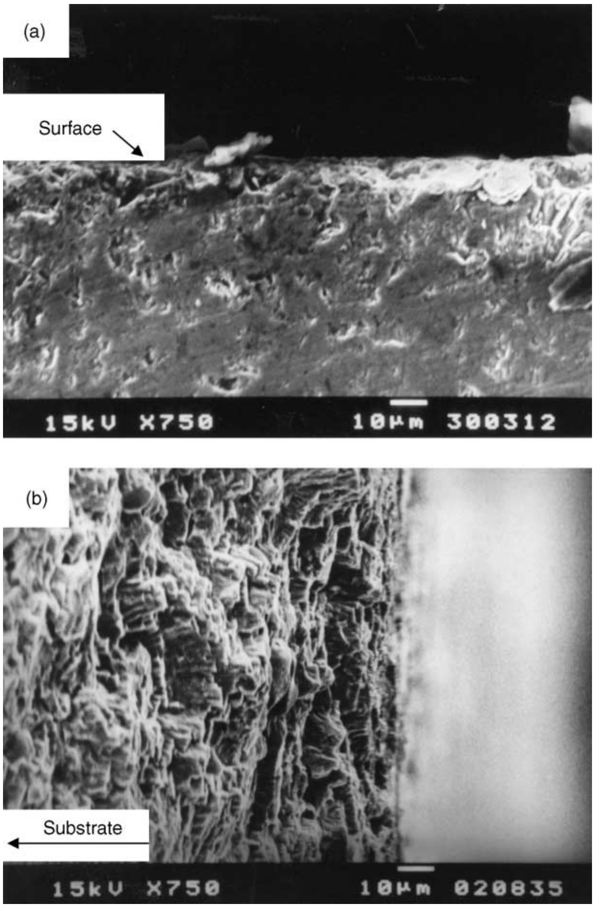


Fig. 9. Typical SEM micrographs of the top layers (Zone 'A') of (a) as-sprayed deposits, and (b) microwave treated spray deposits showing heating effect.

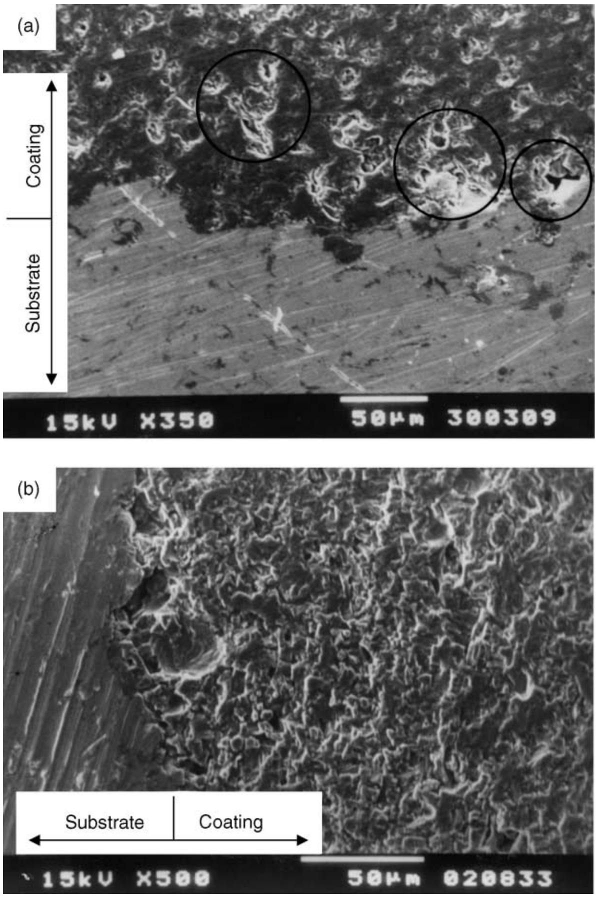


Fig. 10. Typical SEM micrographs of the interface (Zone 'C') of (a) as-sprayed, and (b) microwave treated spray deposits.

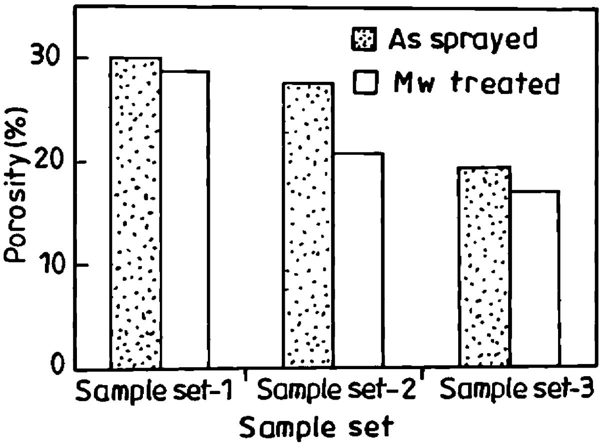


Fig. 11. Typical observed porosities of the ceramic composite spray deposits. (Mw: microwave). ▨, As sprayed; □, microwave treated.

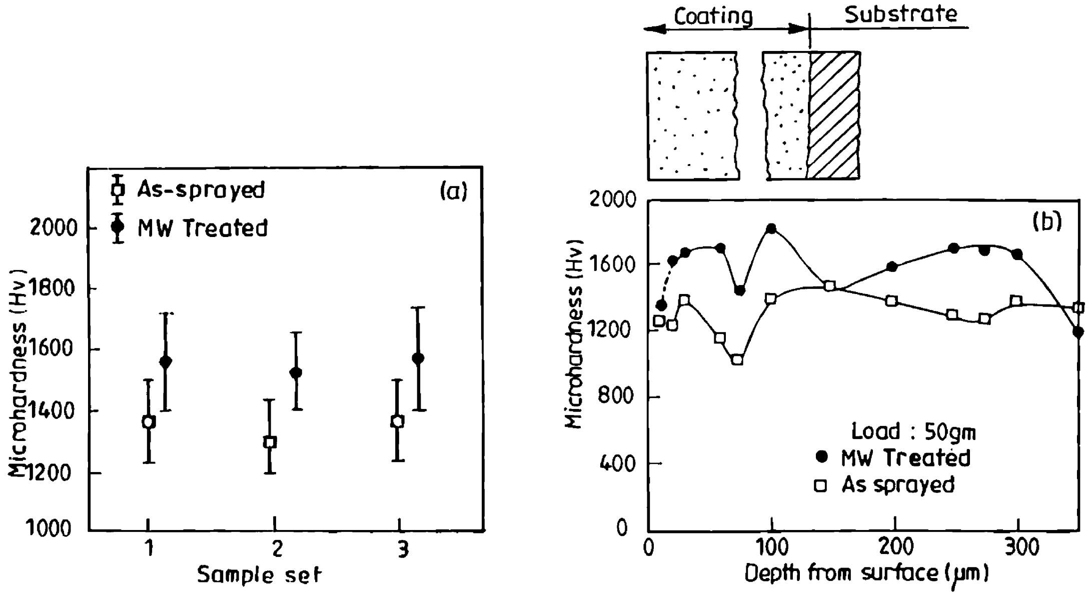
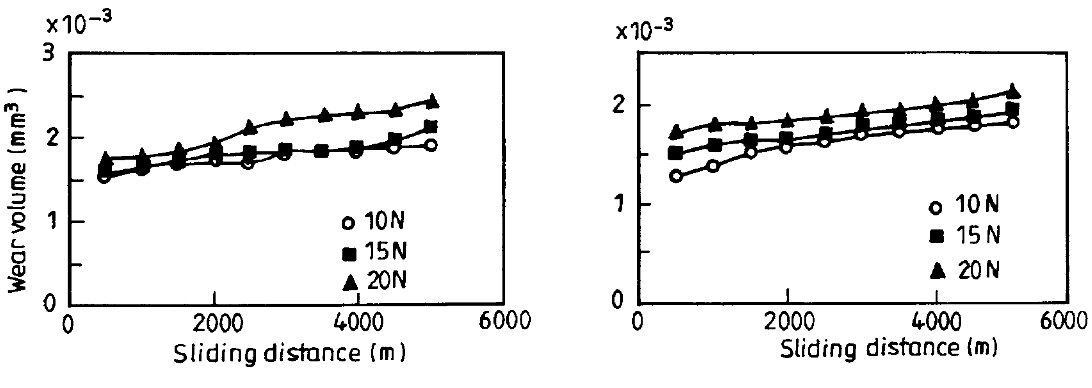


Fig. 12. Observed microhardness of the spray deposits (a) surface, and (b) along the thickness. □, As sprayed; ●, microwave treated.

irradiation. This is due to the instantaneous nature of energy transfer during interaction of microwaves with molecules. As soon as the external electrical field is withdrawn, i.e. the propagation of electromagnetic waves through the material stops, the translational motions of the dipoles, responsible for heating effect, also ceases, resulting in the end of further energy transfer.

4.5. Microhardness survey

Microhardness of the as-sprayed and microwave irradiated spray deposits is illustrated in Fig. 12. Enhancement in microhardness of the post processed ceramic composites is seen along the surface as well as through thickness. This can be attributed to the observed glazing associated with structural refinement and $\alpha \rightarrow \gamma$-$Al_2O_3$ transformation induced densification. The hardness value drops at around 80 micron depth from the surface after an increase from a low initial value, close to the top surface. However, beyond 80 micron depth, there is a sharp increase in hardness values around the core region of the spray deposits, where the layered structure is more compact with highest densification associated with closure of voids and micro cracks owing to reverse gradient during microwave–material interaction. The decreasing trend of microhardness with increasing depth can be attributed to the reduced order of microwave-material interaction owing to the fact that most of the electromagnetic waves are reflected back by the metallic substrate as well as Ni–Al phase. Identical patterns of hardness variation can be attributed to the layered structure of the spray deposits and consequent response to the microwave treatment. However, a reasonable improvement in the microhardness up to a depth of around 350 micron can be seen.



(a) As-sprayed

(b) Microwave treated

Fig. 13. Typical wear volume characteristics of the spray deposits in sliding wear test. ○, 10 N; ■, 15 N; ▲, 20 N.

4.6. Wear resistance

Wear resistance of engineering surfaces is an important performance criterion. Mating surfaces in most of the machine elements undergo continuous degradation owing to gradual wear leading to failure. Turbine blades, impellers, other machine components in the seashore and desert areas experience continued impact of the particles/abrasives resulting in erosion of the exposed surfaces. Critical surfaces of the components in such applications can be protected to a large extent by application of wear resistant coatings on them. However, performance of such coatings depends on number of parameters. This study addresses two important wear phenomena, sliding wear and erosion wear of the coated AT-13 surfaces under different load conditions and are discussed in the following subsections.

4.6.1. Sliding wear

Spray deposited alumina–titania composites were subjected to sliding type of tribocontact in a pin-on-disc arrangement to evaluate their sliding wear resistance.

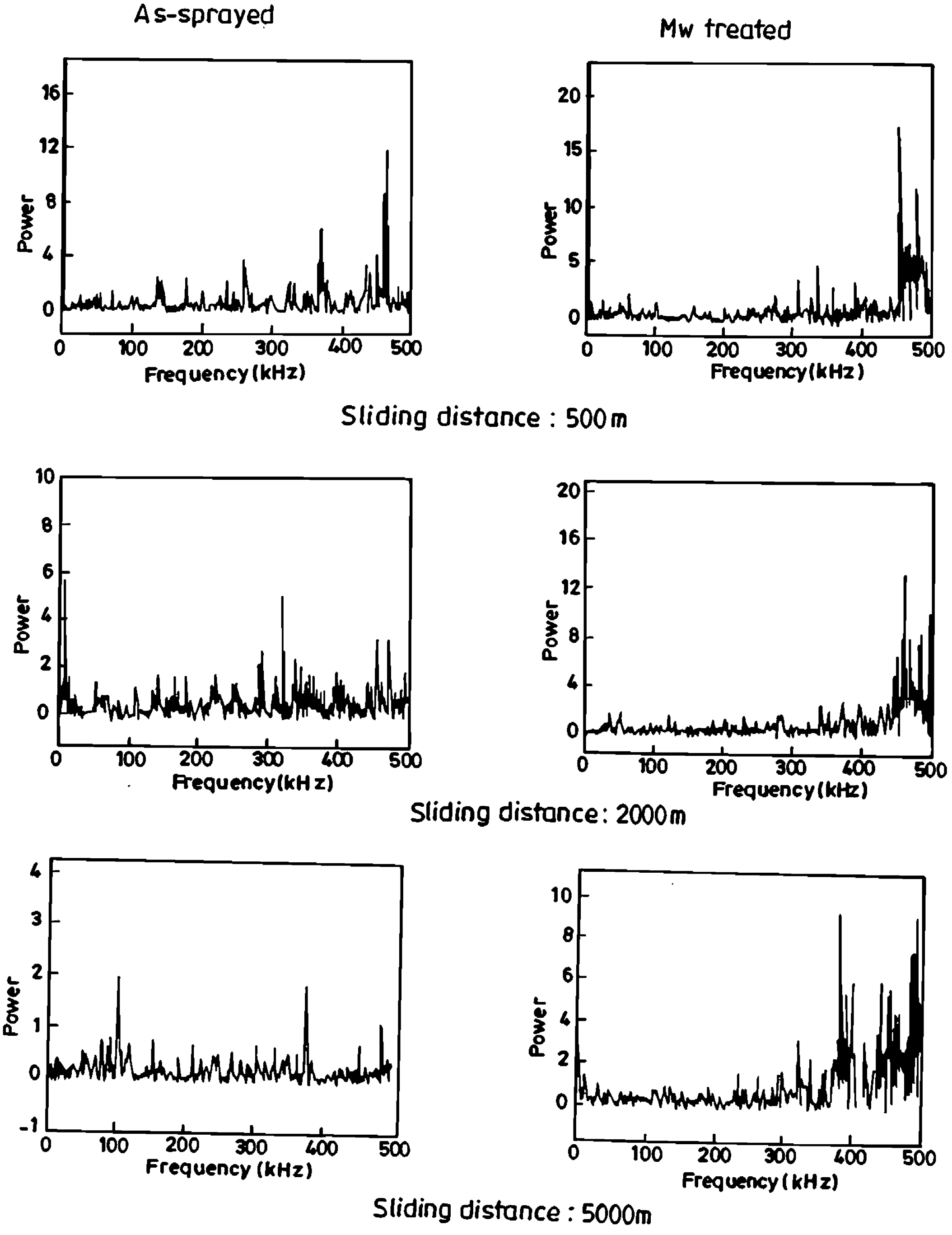


Fig. 14. Typical power spectra of the monitored AE signal at different stages of sliding test. (Load: 15 N). ●, As sprayed; ■, microwave treated.

Typical wear volume characteristics are illustrated in Fig. 13. From the illustration, a general increase in the wear volume with an increase in normal load is observed. Although an increasing trend of wear volume with the increase in load and sliding distance is seen, beyond 2000 m of sliding, a higher order of wear at higher load (20 N) in the as-sprayed coatings is observed. However, in the post treated specimens, a uniform rate of increase in wear characteristics is seen. The observed marginally reduced order of wear volume in the case of microwave treated deposits is attributed to the refinement in grain structure and enhancement in microhardness as reported earlier in Sections 4.2 and 4.5. After 4000 m of sliding, higher order of spalling contributes to further higher rate of increase in wear volume in as-sprayed coatings with localized glazed regions. In the case of microwave treated deposits, as the top layers get worn out and the core region is exposed to the counter face as sliding continues, the rate of increase in wear volume is seen very marginally owing to the observed densification along the depth.

4.6.1.1. Acoustic emission response. Acoustic emissions (AE) emanated during the sliding wear test were monitored for better understanding of material response of the spray deposits. Typical power spectra of the monitored AE signals at different stages of sliding while subjected to 15 N normal load are illustrated in Fig. 14. In case of as-sprayed coatings, a mixed mode of signal with low amplitude is seen. This is associated with initial attritious wear of the ceramic composites. However, in case of microwave treated spray deposits, a higher power signal with dominant high frequency peaks can be seen; this can be attributed to the occurrence of localized rubbing and crest flattening, associated with burst mode of emission during micro cracking of hardened post treated deposits.

4.6.2. Erosion wear

For assessing the effective utilization of microwave irradiation, the as-sprayed and microwave irradiated AT-13 ceramic composites were subjected to erosion wear test through ultra sonic impact using boron carbide abrasive slurry in water medium. Erosion load was maintained by controlling the amplitude of oscillation and slope of the oscillating head. Typical weight loss results are illustrated in Fig. 15. It is clearly seen that the post processed spray deposits exhibit higher order of erosion resistance in terms of lower weight loss in all loading conditions. The initial high weight loss up to around 300 s of erosion impact in all loading conditions is attributable to the removal of the low hardness top layer having higher porosity. This is followed by a sharp drop in weight loss as the impacting particles approach the dense core region of the spray deposits. Twelve hundred seconds and 350 s of continuous impact are observed to be the critical erosion time (t_{crit}) for 5 N and 10 and 15 N load respectively for AT-13 ceramic composite coatings. Erosion resistance drops again at around 1500 s in case of a 5 N load and at around 500 s in case of 10 N and 15 N load while eroding Ni–Al layer is followed by an increasing trend as the eroding particles approach the mechanically interlocked splats with the roughened surface.

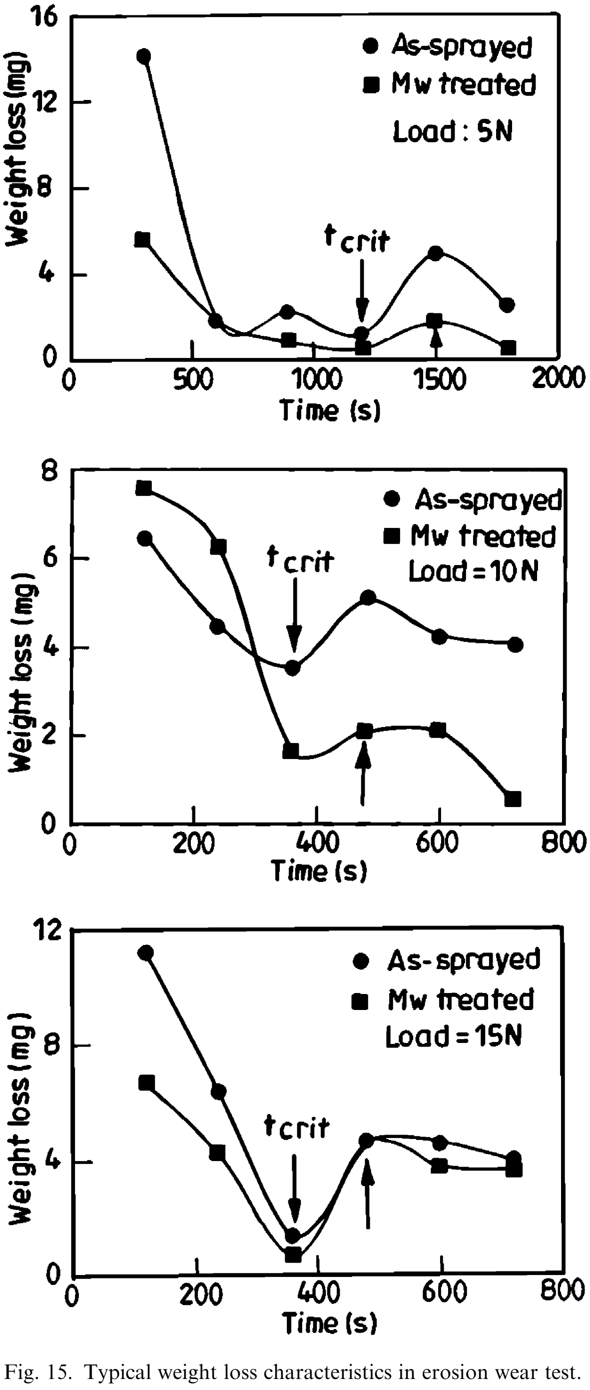
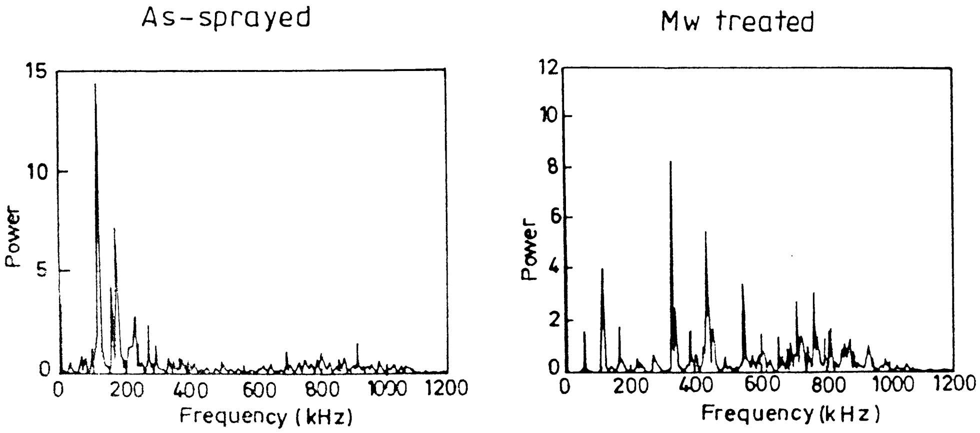
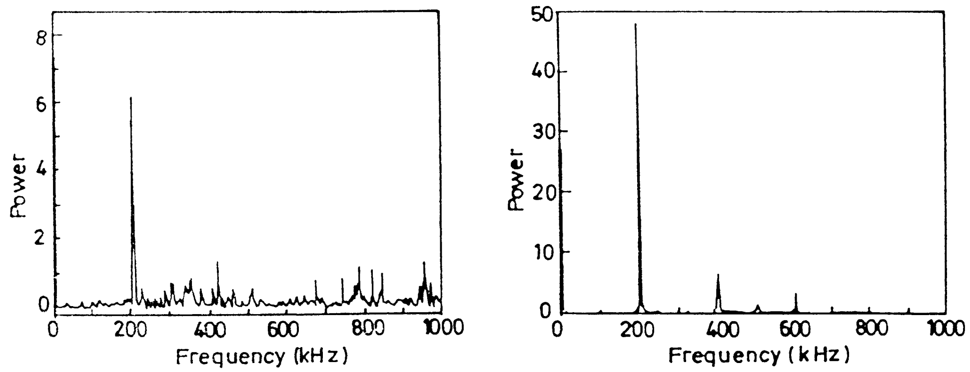


Fig. 15. Typical weight loss characteristics in erosion wear test.

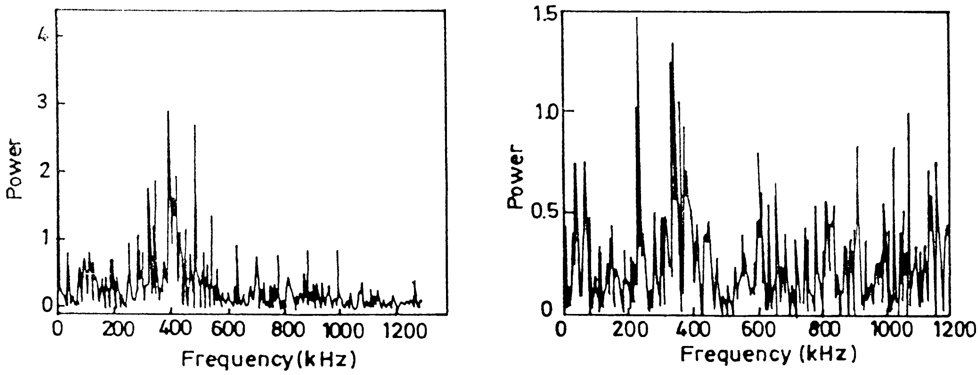
4.6.2.1. Acoustic emission response. To assess the materials response of the spray deposited ceramic composites to the eroding particle impact; acoustic emission signals emanated during the erosion test were monitored. Typical power spectrum of the monitored AE signals at different stages of erosion is presented in Fig. 16. Mostly, a mixed mode of emission is observed in as-sprayed as well as microwave processed spray deposits. High power, low frequency signals at the initial stages of erosion is associated with the removal of low hardness, high porosity top layer of the coatings where particle dislodgement, rather than splat deformation/cracking is predominant. As erosion progresses, dominantly mixed mode, low power signals are monitored. This is associated with the occurrence of a large number of low amplitude AE events in the dense core region. Occurrence of higher power burst signals at around 500 s of impact signifies fracture of the harder splats caused by the impacting abrasives near the core regions. This is followed by the occurrence of mixed mode low power signals indicating the particle dislodgement near the bond coat-substrate region in both as-sprayed and post processed ceramic composites.



After 4 minutes



After 8 minutes



After 12 minutes

Fig. 16. Typical power spectra of the monitored AE signal at different stages of erosion test. (Load: 10 N).

5. Conclusions

Post treatment of plasma spray deposited alumina–titania ceramic composite coatings on steel substrate is successfully carried out using microwave radiation. Microwave treated spray deposits were evaluated through different tests. Preliminary results are reported. There are many issues including determination of core temperature, optimization of irradiation time, optimization of process parameters for particular application, etc. that need to be investigated further. The major conclusions drawn from this study are:

1. Microwave irradiation (microwave glazing) could be an effective post processing technique for plasma deposited ceramic composites.
2. Microwave irradiation induces $\alpha \rightarrow \gamma$-$Al_2O_3$ phase transformation leading to bulk densification.
3. Post processed ceramic composites exhibit enhanced properties in terms of reduced porosity, increased microhardness, and increased wear (sliding and erosion) resistance.
4. Structure refinement of the spray deposits is more dominant in the core region of the ceramic composite due to reverse thermal gradient.
5. Acoustic emission monitoring could be effectively employed to assess the materials response of the as-sprayed as well as microwave post processed ceramic composite.

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