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Continuous Microwave Plasma Processing of Cold Drawn Steel Wire Rod

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Abstract— **Surface modification of cold drawn steel wire rod has been performed using a set of microwave plasma torches operating at 2.45 GHz, 3 kW maximum power, with the aim to modify the wire surface morphology and chemical composition prior to hot dip coating with Zn-based alloys. The effect of varying the carrier gas type and flux is investigated experimentally and by numerical simulation. Adhesion of the zinc-based coating after plasma treatment, compared to untreated samples, as well as corrosion resistance, resulted improved, however treatment reproducibility can be still considered poor**

Keywords: plasma torch, numerical simulation, metallurgy, surface engineering,

I. INTRODUCTION

Wire drawing of steel consists in a series of subsequent section reduction, starting from a wire rod, with intermediate annealing thermal treatments in order to counter the effects of cold working, and to allow more further drawing. Commercial wire drawing usually starts with a coil of hot rolled wire, whose surface is first mechanically or chemically treated to remove scales. It is then fed into continuous wire drawing machine. When the final shape is reached, the wire surface is contaminated by the lubricants used during drawing operations, and by possible oxides formed due to the annealing treatments.

This surface contamination must be removed before any protective coating, like zinc dip coating, can be applied. This is usually accomplished by chemical methods (hot pickling with hydrochloric and sulphuric acid water solutions), electro-chemical methods (in diluted sulphuric acid water solution and use of current) or by the less effective mechanical methods (forced deformation in passing trough dies or by series of bending operations). Then, to maximize adhesion of the coating and favour reactions with the steel substrate, the surface need to be prepared, typically by ammonium chloride treatments and other fluxes and oxide-inhibiting compounds. The removal of surface contamination could be favorably accomplished also by the use of plasma, as recently reported in case of aluminium alloys [1], showing that with a proper control of the process parameters, like electric field value and pressure level, cleaning of the wire can be achieved.

Microwave induced plasmas possess some important benefits, such as the high efficiency in generating chemically active species, relatively high electron density [2], fast and nearly contamination-free processes and capability of operating in a wide pressure range [3]. One of the main technical difficulties in implementing this technique, is the need of a proper matching, able to account for the impedance variations of the load, deriving even by small changes in the plasma characteristics. A proper design of the microwave plasma source could significantly reduce the sensitivity of microwave systems to load variations, and the knowledge of the possible system statuses could help designing and implementing a simpler, faster and cheaper automatic impedance matching device (simply switching between a fixed number of possible stares) without significantly affecting the plasma homogeneity.

Existing electromagnetic field modelling software can help the designer in accounting for such rapid variations, especially when direct measurements or existing applicators are not possible or available. However, it is necessary to describe plasma as a load in the numerical simulation, and this usually poses problems because it requires complex fully coupled thermodynamic, fluid-dynamic and electromagnetic models. A possible alternative, at the microwave frequencies, is to represent plasma like a dielectric, since microwaves interact with plasma in the quasi-optical manner, i.e. not with the single charge carriers but in collective regime [4]. A variation of the plasma characteristics, thus, can be represented by a variation of its equivalent permittivity, and implemented in the model.

Aim of this work is to present the design of a microwave atmospheric plasma torch and the preliminary results obtained in processing cold drawn steel rod. This new treatment, for iron-based alloys, besides providing cleaning, is expected also to perform simultaneously heat treatments (annealing, thanks to the high temperature and high heat transfer coefficient from the plasma torch to the wire) and surface activation, in a single step.

II. NUMERICAL SIMULATION

Numerical simulation of the plasma torch was limited to the electromagnetic field distribution in the applicator and the minimization of reflected power, i.e. without considering the thermal and fluid-dynamics aspects of plasma generation. The software concerto 3.5 was used to perform the numerical simulation of the plasma torch, whose design is based on a WR340 waveguide with two ports in cutoff conditions for processing gases inlet and outlet. Figure 1 shows a rendered image of the model. Only the final part, i.e. the applicator, was modeled, including a waveguide portion, a waveguide to coaxial line transition and a cylindrical tube for plasma generation and containing.

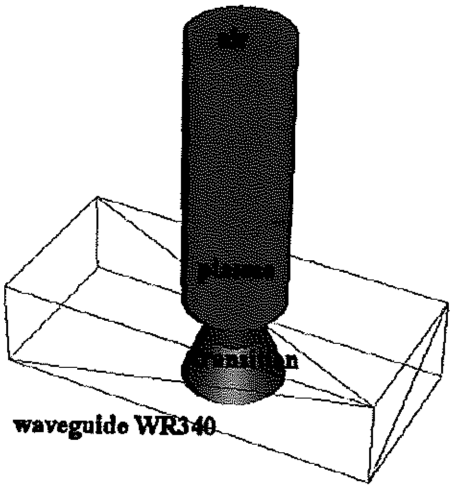

Figure 1. Base geometry of the microwave applicator.

In case of microwave plasma sources, there are two main statuses of the system, completely different: before plasma ignition, corresponding to the "empty applicator" condition, and after plasma ignition, corresponding to the "loaded applicator" condition. Since the ignition and sustaining of plasma, especially at high pressure, depend on high electromagnetic field intensity [5], this condition must be fulfilled in both system statuses. Moreover, after plasma ignition, pressure, nature of gases and electron temperature can vary with time, and this phenomenon is equivalent to a change in the dielectric properties of the load.

The load, which is represented by the plasma itself, can be described in terms of its complex permittivity, according to[3]:

$$\varepsilon^* = 1 - \frac{\left(\varpi_p/\varpi\right)^2}{1+\left(v/\varpi\right)^2} - j\,v/\varpi\,\frac{\left(\varpi_p/\varpi\right)^2}{1+\left(v/\varpi\right)^2} = 1 - \frac{\frac{n_e e^2}{\varepsilon_0 m_e}\left(1+j\frac{v}{\varpi}\right)}{\varpi^2+v^2}$$

where n_e= electron number density, e= elementary charge, m_e= electron rest mass, ω is the excitation frequency, ω_p is the plasma frequency and v is the collision frequency between electrons and neutrals, which is a function of the pressure, the gas nature, the gas temperature and the electron temperature [3]. Any change in one of these variables induces a change in the plasma equivalent permittivity, and thus in the load impedance, which needs to be adjusted by a proper impedance matching device.

Since, depending on the parameters of operation of the plasma torch (gas type, gas flux, pressure, microwave forward power), permittivity can vary significantly, especially in its imaginary part, numerical simulation was aimed at finding the applicator geometry able to provide the proper conditions for plasma generation and sustaining as well as able to withstand severe load variations without requiring automatic impedance matching devices to retain a satisfactory energy efficiency at 2.45 GHz. Hence, the objective function for optimization was that |S11| remained lower than 0.2 for the expected load variations. Complex permittivity of the load (plasma equivalent dielectric) have been varied over 2 orders of magnitude to take into account possible variations of processing parameters, and the applicator geometry modified accordingly.

Figure 2 shows the simulated reflection coefficient value (|S11|) as a function of frequency for three different loading conditions.

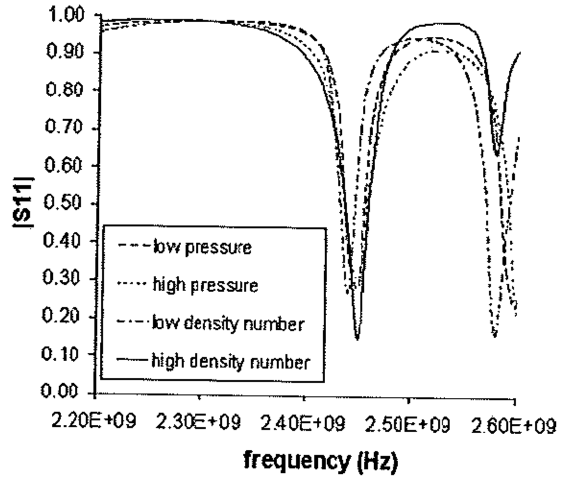

Figure 2. variations of |S11| as a function of plasma properties. Pellet

Despite the satisfactory impedance matching, the minimum value of |S11| occurs only on a narrow bandwidth; moreover, the original geometry suffers from the small space available to perform the plasma treatment on the passing wire rod. Thus, a second geometry has been investigated, with a tapered output section and a tuning stub. The new geometry is depicted in figure 3, showing also the door-knob transition used in the waveguide to coaxial junction.

Numerical simulation results allowed to find the most efficient doorknob transition dimensions, according to the data plotted in figure 4, referred to the minimization of the reflection coefficient in the 2.44-2.46 GHz frequency range (objective function). In this case, the average value of the reflection coefficient calculated at 2.45 GHz results 0.28 in case of low electron density number and 0.19 in case of high electron density number.

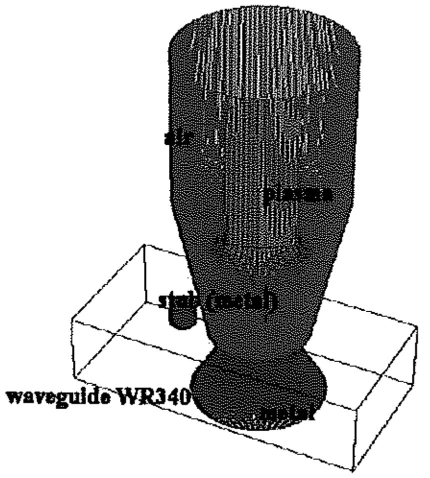

Figure 3. Close up view of the microwave applicator and door knob transition

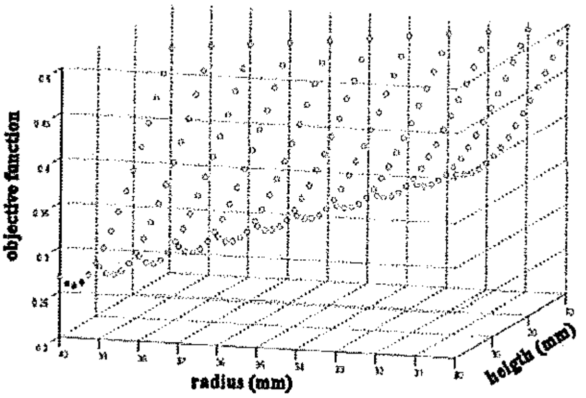

Figure 4. optimized doorknob transition dimensions

In order to avoid any possible unwanted microwave emission from the tapered transition, in case no plasma is present, the upper part of the applicator has been closed by a furthered tapered section, with progressively smaller radius, until the achievement of the cutoff conditions. Thus, the double-tapered upper part allows an increase of the dimensions of the plasma generation zone, while preventing any microwave leakage.

III. RESULTS AND DISCUSSION

Based on the numerical simulation results, a prototype plasma source was built and installed on an Alter TM030 microwave generator, able to provide a maximum RF power of 3000 W at 2.45 GHz. The microwave applicator was realized modifying an existing Sairem downstream source, installing the waveguide to coax doorknob transition by means of the upper and lower round flanges of the original applicator.

Preliminary tests were conducted in order to verify the good impedance matching of the original design, as well as the absence of microwave leakage. Figure 5 shows the experimental setup during Ar plasma processing, with the microwave leakage detector in close up. The measured reflection coefficient, by means of a directional coupler, resulted higher than calculated by numerical simulation: power reflection amounted to 13±9%. This is probably due to the approximation of the plasma as the equivalent dielectric and to small variations ascribable to plasma instability.

Further tests have been conducted using the prototype torch and treating metal blades artificially covered by lubricating oil. The selection of such large samples allowed to measure the surface temperature by optical pyrometers, and thus verifying the possibility of simultaneously performing cleaning and annealing treatments. Figure 6 shows the sample during processing and, in the box, a close up view of the sample after 5 seconds exposure to the plasma.

Figure 5. Ar plasma torch and microwave leakage detector

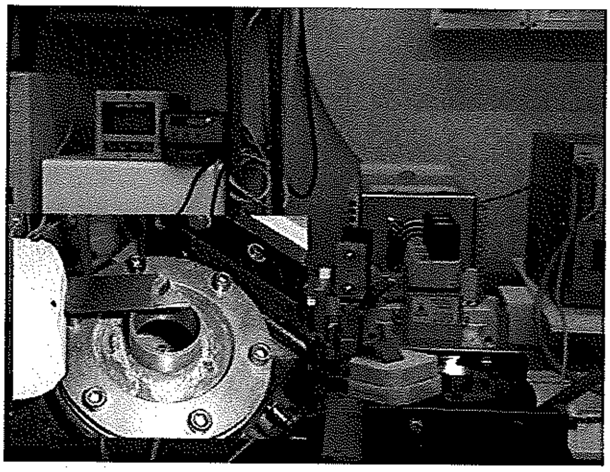

Figure 6. plasma processing of an artificially oil-contaminated cutting blade

Temperature measured during processing reached 905°C and the plasma torch efficiently removed the surface contamination. However, due to the unprotected processing atmosphere, oxidation of the surface not directly exposed to the Ar plasma occurred.

Subsequent tests were conducted on 2 mm thick steel wire, after approximately 15 days since its last annealing step on the Trafileria e Zincheria Cavatorta plants in Calestano (Italy) and stocking in closed boxes. The wire was continuously passing trough the plasma generation zone at about 1 meter per minute, without temperature control. Results confirmed the possibility of performing plasma cleaning of the surface, even if some contaminated regions remained in localized spots of the wire. This can be ascribed once again to the plasma instability, as well as to the fact that treatments are performed in open air, and disturbance to the plasma torch are induced by air motion in the surrounding space. Moreover, some of the samples presented a pronounced and not homogenous oxidation, which was only partially affected by the plasma treatment. As a matter of fact, some darker colored spots were still detectable visibly after plasma torch processing. The preliminary tests suggest the need to operate in closed and controlled atmosphere conditions, a solution which will make the final process slightly more complex but will avoid unwanted contamination and lack of reproducibility.

Nevertheless, a series of plasma-treated samples subjected to hot zinc dip coating operations, using a Zn-Al alloy, was produced, and adhesion of the zinc, measured by scratch test at progressive load, evidenced a much higher adhesion with respect to untreated wire. Table I summarizes the scratch tests conditions and results, averaged on 5 scratches in different positions along the longitudinal direction of the wire.

TABLE I. SCRATCH TESTS CONDITIONS AND RESULTS

	Plasma treated	Untreated
Scratch length	3 mm	3 mm
Indenter	Rockwell, 100 µm radius	Rockwell, 100 µm radius
Scratch speed	1 mm/min	1 mm/min
Critical Load	17.2 ± 1.9N	8.3 ± 0.4N

The poorer reproducibility of plasma treatment is evident also from the high scattering in scratch resistance, compared to untreated samples. Better results could probably be achieved pre-cleaning the wire in order to achieve a partially de-oxidised and de-scaled wire, since the small time of exposure to the plasma torch is not enough to completely clean the steel surface, in the experimental conditions tested.

IV. CONCLUSIONS

In this work, a microwave plasma torch, operating at atmospheric pressure, was designed and built. Numerical simulation results evidenced the need to account for drastic equivalent load permittivity variations based on the plasma characteristics affected by processing variables like microwave forward power, gas type and flux. Fixed impedance matching device systems were installed and experimental testing demonstrated the possibility of achieving a satisfactory low power reflection (less than 15%). Impedance matching for a given load characteristic, instead, allowed to practically nullifying reflected power, but increased sensitivity to load variations.

Using an Ar-plasma torch, it was possible to clean contaminated surfaces of steel wires, while heating the bulk material in excess of 900°C. The heavily oxidized parts resulted only partially treated, suggesting the need of a mechanical pretreatment to descale the wire. However, in the manufacturing line, wire exiting last annealing in controlled atmosphere is only mildly oxidized, and such preliminary treatments could be avoided. Despite such problems, preliminary tests with hot zinc dip coating evidenced a strong increase of zinc adhesion to the substrate steel in case of plasma processing with respect to untreated surfaces. Further investigations are in progress, concerning both microstructural modifications of the plasma-treated steel and the effects of plasma cleaning immediately after the last annealing step in the wire drawing line.

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