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FLAME-SINTERED CERAMIC EXOELECTRON DOSIMETER SAMPLES

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ABSTRACT New techniques for the preparation of integrating solid state dosimeters, particularly exoelectron dosimeters, have been initiated. The procecure consists in melting the powdered dosimeter materials in a hot, fast gas stream and depositing the droplets onto a sample substrate, thus sintering a sensitive ceramic layer. The gas stream is generated either through a chemical flame or by an electrical arc plasma. Results will be reported on the system Al_2O_3/stainless steel as a first step to a usable exoelectron dosimeter.

1. Introduction

The deposition of thin ceramic layers by oxy-acetylene flame-spraying of powders, particularly Al_2O_3, has been introduced in engineering already more than twenty years ago /1/. The main purpose to develop this technique was surface protection of metallic parts against corrosion /2/. Lateron this method was improved by adopting plasma-flame technology /3/. Since then, even more sophisticated applications have been envisaged, for example in the production of microelectronic devices /4/. Recently, flame-spraying has been applied also in the preparation of integrating solid state dosimeters, particularly exoelectron dosimeters on the basis of aluminum-oxide /5//6/.

2. Chemical- and plasma flame spraying processes

The apparatus' for chemical-respectively plasma flame spraying differ considerably in size (there is a factor of 10 in price). The simple chemical flame gun is very practical for initial laboratory work, whereas the plasma gun is also appropriate to industrial scale application.

In our laboratory we used a LURGI-GOTEK gun as schemed in Fig. 1. Oxygen and acetylene are mixed inside the gun and react in the flame which is ejected from a nozzle. The powder, coming from a vibrator feed, is injected axially into the flame by a separate oxygen gas stream. The chemical flame reaches a temperature of 2000 °C at 2 cm from the nozzle. Attaining a speed of approximately 100 ms the powder grains are molten within a few milliseconds. The distance of the substrate is optimum at approximately 10 cm.

In cooperation with the SFEC+) Corporation a plasma gun of the type METCO 2 M B /7/ was disposable, Fig. 2. The plasma flame originates from an electrical arc burning inside a chamber and forcing out a plasma gas (nitrogen-argon) through the nozzle as a flame. By a large electric energy consumption (up to 80 kW) temperatures up to 15 000 °C and velocities up to Mach 2 are reached. The powder is injected at the periphery of the nozzle and projected to the substrate which is located in a distance of about 20 cm. The time of flight is in the submillisecond range.

3. Powder and layer characteristics

The grain size of the original powder particles shall be in the order of 10 µm to 50 µm for chemical flame spraying; for plasma flame spraying particles up to some 100 µm can be used. In both cases the grains are molten into small spherical droplets which are quenched at the cold substrate. The layer is established by welding successively one particle onto the others. Thus a certain porosity is incorporated. Even in the case of plasma deposition, which is expected to produce rather dense layers by the large particle impact, the porosity is not lower than 10 % of the total layer volume /8/. Large inner surfaces are favorable in order to obtain a low electrical resistance even for ceramic layers with a thickness of some 100 µm /6//9/. These can be fairly good reproduced already by chemical flame spraying. In principle, for the preparation of exoelectron dosimeters, single grain layers would be already sufficient, since the electron emitting surface layer amounts only to a small fraction of the grain size. Very thin ceramic layers are prepared preferably by plasma flame spraying.

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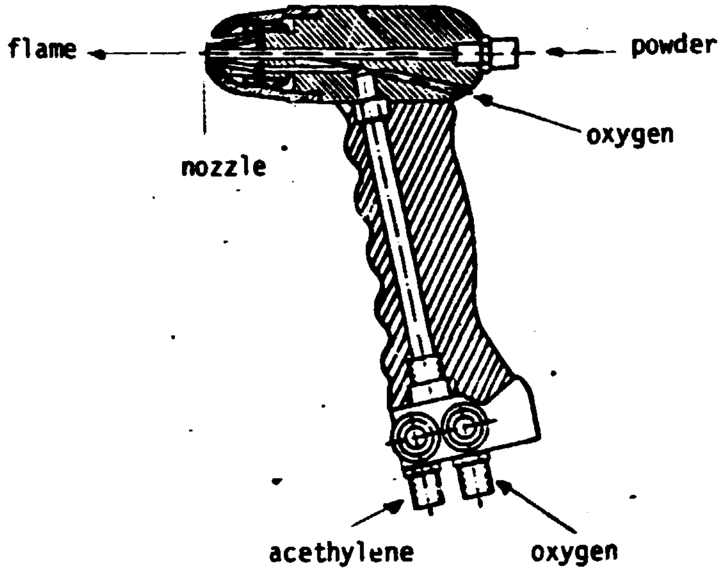

Fig. 1 LURGI-GOTEK chemical flame spray gun /2/

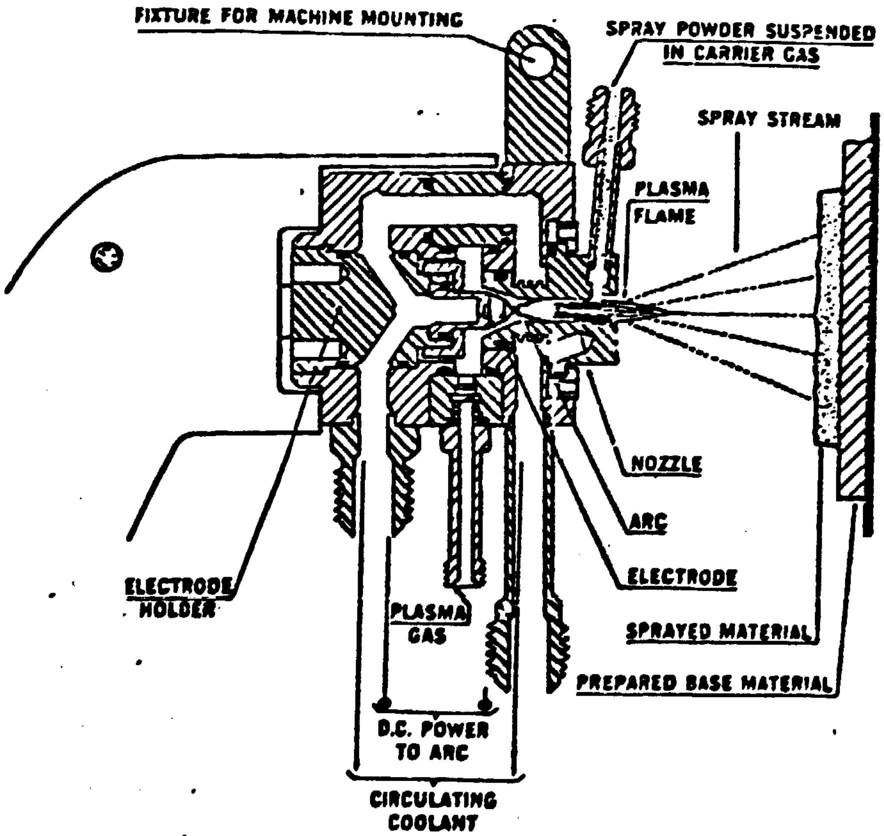

Fig. 2 METCO Type 2 M B plasma flame spray gun /7/

4. Preparation and propeties of exoelectron dosimetry samples

In this initial stage of adopting flame spray techniques the well-proved combination of stainless steel substrates with alumina (chemical grade Al_2O_3 MERCK Art. 1095) was mainly used for the preparation of exoelectron dosimeter samples. Chemical flame sintered (CFS-) layers adhere rather firmly; plasma flame sintered (PFS-) layers can be removed only by sample destruction.

After preparation, the samples exhibit without additional excitation a thermally stimulated electron emission being much stronger from PFS- than from CFS-layers. This spurious signal is removed by preannealing for at least 1 hour at 700 °C in air. Afterwards, only very small tribosignals are induced by occasional scratching the sample surface.

Irradiating by a Sr^{90} β-source or a 10 kV X-ray source and reading the samples in a CH_4-flow counter resulted in the glow curves of Fig. 3. Here, PSF-layers show glow curves which are nearly identical with those of the original, sedimented powder /10/, whereas CFS-layers tend to pronounce the front part of the high temperature double peak used in dosimetry.

The sensitivity of the samples against radiation as defined by the integral number of electrons counted per emitting area and dose differs considerably. Optimum values of 700 $e/mGy \cdot cm^2$ for CFS-layers and 1200 $e/mGy \cdot cm^2$ for PSF-layers were obtained with a background of 200 e/cm^2. The sensitivity of sedimented Al_2O_3 powder, 5000 $e/mGy \cdot cm^2$, is not yet reached. The dose response of the samples proved to be always linear over at least 3 orders of magnitude, see also /6/. Reproducibility measurements showed a standard deviation of ± 3 % for single detector samples and of ± 10 % for samples of the same badge, equally for CFS- and PSF-layers.

The intra-badge reproducibility, which is not yet sufficient, depends strongly on the constancy of the flame spray parameters (e.g. composition, speed, temperature of flame, layer thickness, grain size). In the same way, the stationary (thermionic) emission, which superimposes the exoelectron emission in the dosimetry peak, is not always kept sufficiently low.

The quality of the dosimetry samples is further impaired by an unwanted effect: The initial, exoemissive α-Al_2O_3, as produced by calcination of γ-Al_2O_3 /11/, is partially retransformed in the flame to the non-emissive γ-Al_2O_3 modification with a changing α/γ-Al_2O_3 ratio.

Improvement of sensitivity and reproducibility is expected by converting the α/γ-Al_2O_3 completely to α-Al_2O_3. This can be performed by flame spraying on a hot substrate /12/ or by postannealing the samples at 1200 °C. /8/. Both

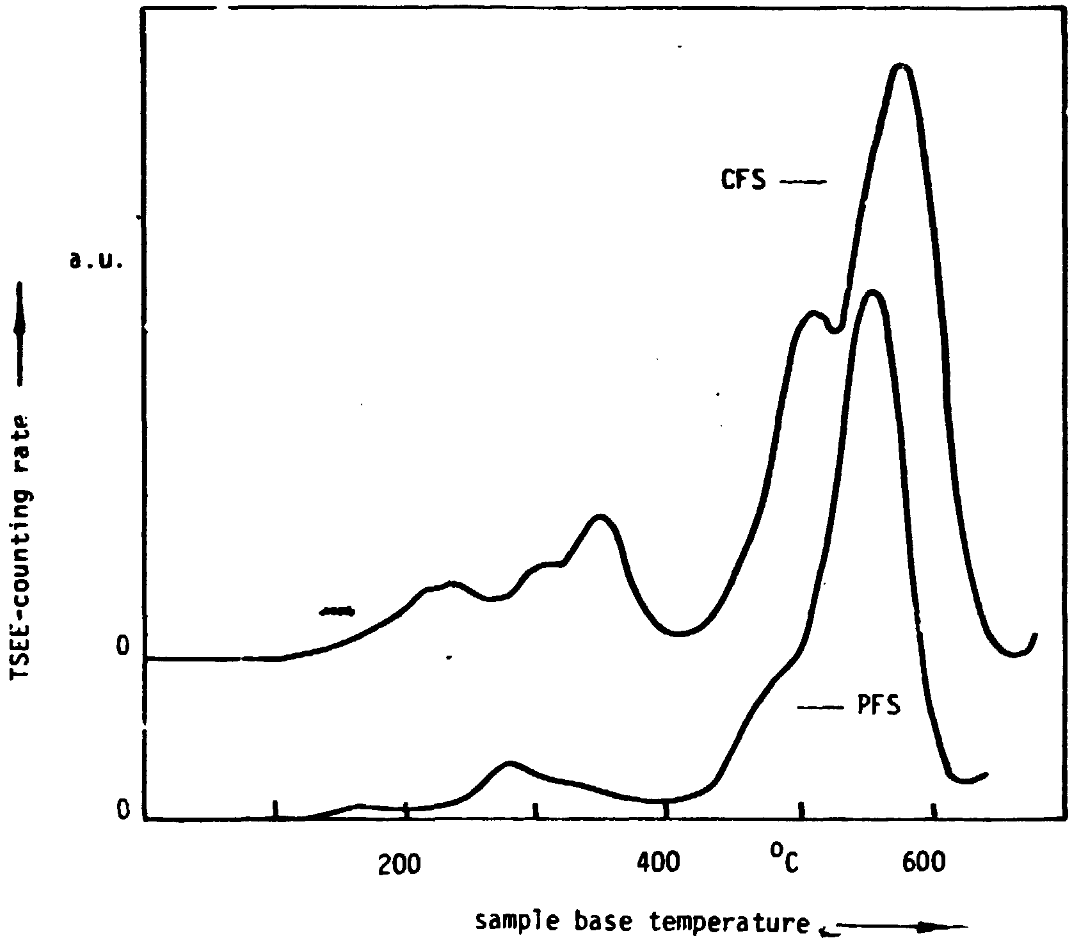

Fig. 3 Thermally stimulated exoelectron glow curves of Al_2O_3 after chemical flame spraying (CFS) and plasma flame spraying (PFS). Heating rate 1 K/s

procedures require inert substrates. Experiments have been commenced now with graphite discs, which are, on the other hand, also interesting for dosimetry owing to their low atomic number.

Acknowledgement

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