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# Residual stresses and texture in Ni/SiC nanocomposite coatings

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## Abstract

Nanocomposites of Ni–SiC were electrodeposited on a 2024-T3 aluminum substrate in a nickel sulfamate bath. The goal of the study was to determine the effect of the SiC particle size and concentration on the crystallographic texture and residual stresses that develop in the nickel matrix. The presence of crystallographic texture, its nature, and its intensity affect all mechanical and physical properties of the coating. Residual stresses, if highly tensile, may be detrimental to the performance of the nanocomposite coating. The texture and residual stresses evolving in the coatings were studied using X-ray diffraction. Composites with nano-particles developed a strong  $\langle 1 0 0 \rangle$  fiber texture and stresses that were compressive in small SiC concentrations, but tensile with low magnitude at greater concentrations. The composite with coarser SiC (500 nm) demonstrated a very weak  $\langle 1 1 1 \rangle$  texture and residual stresses that are tensile in nature. Based on the findings, nanocomposites are expected to perform better than micro-composites that were electroplated using the same coating parameters.

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# 1. Introduction

Nickel coatings have been used in numerous applications for many years. Their uses range from mechanical (preventing wear) and chemical (protecting against corrosion) to electrical and magnetic (storing electronic data in magnetic media) [1,2]. For applications of coatings that take advantage of the mechanical properties of nickel, blending hard nano-particles into the coating offers an additional increase in strength. The goal in producing metal-matrix nanocomposite coatings is to obtain highly wear-resistant, strong, thin coatings that adhere strongly to inexpensive substrates. They show potential for use in many fields, such as in the aerospace and automotive industries [3,4]. A variety of techniques may be used to produce such coatings, but electroplating is simple and economical [5]. In addition to their contribution to the composite properties, the nano-particles cause change in the nickel matrix structure (e.g., grain size and crystallographic texture) [6].

Residual stresses often accompany electroplating. They can result from many sources, including crystallographic mismatch, or thermal expansion differences between the substrate and coating [7]. They may be compressive or tensile, depending on

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the coating, the substrate and the process parameters. Tensile stresses are undesired as they may cause a decrease in fatigue life, performance in corrosive environments, and delaminating. Although stress-relief heat treatment may alleviate the situation in the coating, it may adversely affect the properties of the substrate and cause distortion.

The presence of a strong crystallographic texture in a material may affect all its properties (e.g. mechanical, chemical and magnetic) and make them anisotropic [8]. The texture present in coatings is fiber texture because of the planar isotropy of the coating process, but the preferred direction ("fiber") will vary as a function of coating conditions [7].

In the present study, nanocomposite coatings of nickel with silicon-carbide particles were examined for their residual stresses and texture development, and the way these are affected by the SiC particle size.

### 2. Experimental

### 2.1. Coating

Nickel–SiC MMC were electrodeposited onto an aluminum substrate in a nickel sulfamate bath. Aluminum (2024-T3) was selected as the substrate because of its good strength-to-weight ratio, and its need for surface protection in hot and/or abrasive conditions. The coating bath was maintained at a pH of  $4.5 \pm 0.2$  and a constant temperature of  $55 \pm 3$  °C. All the coatings were deposited at a voltage of about  $3.0 \pm 0.3$  V and a direct current density of

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Fig. 1. 500× Al substrate, Ni coating ( $\sim$ 44 µm is observed).

35 A/dm<sup>2</sup> for 15 min; these parameters were chosen to replicate optimal coating results that we had achieved earlier [6]. To study the effect of particle size on properties, three sizes of silicon carbide particles were tested: 20, 50 and 500 nm. Concentrations of SiC in the sulfamate bath were 30, 133 and 400 g/L. Silicon carbide nano-particles were carefully dispersed into the bath. The bath was agitated continuously using an in-house-designed "bubblator" that generated air bubbles into the bath solution at a rate of ~2.8 m<sup>3</sup>/h.

#### 2.2. Testing

The thickness of the deposited layer was measured on a metallographic cross section using an optical microscope (Zeiss MC 80 using the Axio Vision 3.0 software).

Measurements were done at a magnification of  $500 \times$ . The thickness was determined to be around 50  $\mu$ m; see, for example, Fig. 1.

The incorporation of particles in the coatings was determined by atomic force microscopy and scanning electron microscopy, as described elsewhere [9].

X-ray diffraction was used to determine both the crystallographic texture and the residual stress developed in the process. Experiments were conducted on a Philips-X'Pert machine with APD software. A copper anode with a graphite monochromator was used ( $\lambda = 0.1542$  nm).

The two-angle method was used to determine the residual stresses,  $0^{\circ}$  and  $45^{\circ}$  tilt. For each sample, three measurements were done—longitudinal, transverse and at  $45^{\circ}$ . The  $2\theta$  angle was about  $122^{\circ}$ , which corresponds with nickel's  $\{400\}$  set of planes. This angle was chosen because it is high enough to attain the accuracy necessary, and it is parallel to the set of planes  $\{h00\}$  shown previously [6] to lie parallel to the sheet plane. Slow scans with a step size of  $0.03^{\circ}$  and data collected at 10 s per step were used for optimum determination of exact peak position. The position was calculated using the APD software.

The type and strength of the texture in the samples were estimated using regular X-ray scans, by comparing the relative intensity of the  $\{111\}$  and  $\{200\}$  peaks and comparing the results to those in a randomly oriented standard.

# 3. Results and discussion

## 3.1. Texture

The fiber-texture of the nanocomposite samples was a strong  $\langle 100 \rangle$ , see for example Fig. 2 with the  $\{111\}$  and  $\{200\}$  peaks for the sample with 30 g/L of 50 nm particles. The measured  $I_{111}/I_{200} = 0.2$ , as compared with  $I_{111}/I_{200} = 2.4$  for a randomly oriented polycrystalline (based on ICDD data [10]). This result is in agreement with textures we have previously observed [6] using both pole-figures and regular scans. The  $\langle 100 \rangle$  fiber tex-



Fig. 2. The  $\{1\,1\,1\}$  and  $\{2\,0\,0\}$  diffraction peaks of nickel for the nanocomposite with 30 g/L of 50 nm SiC particles. A strong  $\langle 1\,0\,0 \rangle$  texture is demonstrated.



Fig. 3. The  $\{1 1 1\}$  and  $\{2 0 0\}$  diffraction peaks of nickel for the nanocomposite with 30 g/L of 500 nm SiC particles. A weak  $\langle 1 1 1 \rangle$  texture is demonstrated.

ture develops when the growth mode is lateral—"free growth" [11]. It occurs when the stresses are low [12].

The sample with 500 nm SiC particles demonstrated a very weak  $\langle 1 \ 1 \ 1 \rangle$  texture, with  $I_{111}/I_{200} = 2.7$ , as compared with 2.4 for a randomly oriented polycrystalline (Fig. 3).

#### 3.2. Residual stresses

The Young's modulus of the coatings was estimated using tensile testing of the substrate and coating, and applying the law of mixtures. However, due to the inadequacy of that assumption and inaccuracies in measurement of such a sample, the results will be presented here as principal strains ( $\varepsilon_2$  and  $\varepsilon_2$ ). Using the equation  $\varepsilon_1$ ,  $\varepsilon_2 = \frac{1}{2}(\varepsilon_x + \varepsilon_y) \pm \frac{1}{2}\sqrt{(\varepsilon_x - \varepsilon_y)^2 + \gamma_{xy}^2}$ , the results for  $\varepsilon_1$  are presented in Fig. 4 and for  $\varepsilon_2$  in Fig. 5. It can be seen that at low particle concentrations, the highest (tensile) stresses develop in coatings with the relatively coarse



Fig. 4. Principal strain  $\varepsilon_1$  as a function of concentration and particle size.



Fig. 5. Principal strain  $\varepsilon_2$  as a function of concentration and particle size.

500 nm size particles. The composite coatings with particle size in the nano-scale produced compressive strains at low concentrations. With the increase in particle concentration, the residual strains in samples with nano-particles became tensile, but their magnitude remained less than in the low-concentration sample with coarser particles, even when the particle concentration of the nano-scale particles is 400 g/L. This is an advantage of the smaller scale particles.

It may be concluded that the texture and the residual strain results are in agreement. The  $\langle 100 \rangle$  fiber texture, observed in the coatings with sub-micron particles, is typical of the lateral growth mode [11] which is associated with low stresses [12]. The strong presence of  $\langle 100 \rangle$  in the fine-particle composite and its absence in the coarse-particle composite correlate well with a free growth mode that results in lower stresses in the nanocomposites.

# 4. Summary

Incorporating particles of SiC in nickel coatings is done to improve their mechanical properties. In addition to the contribution of the properties of the hard particles to the composite, they also alter the structure and properties of the nickel matrix. In our prior work, the effect of the incorporated particles on grain size was established. In our current work, it was established that the SiC particle size affects both the crystallographic texture and residual stresses of the nickel matrix. The larger, sub-micron particles impeded the lateral growth of the nickel layer, resulting in tensile residual stresses, and a weak  $\langle 1 \ 1 \ \rangle$  texture. The finer particles at the nano-scale result in compressive stresses at low concentrations, but in tensile stresses at higher concentrations, with magnitude increasing with concentration. No significant difference was observed between the effects of the 20 and the 50 nm particles on the nickel matrix.

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