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TECHNICAL REPORT ARCCB-TR-98004

**GRAIN ORIENTATIONS IN  
ELECTROLYTIC HIGH CONTRACTION  
AND LOW CONTRACTION CHROMIUM DEPOSITION**

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## INTRODUCTION

Electrolytic chromium has many industrial and ordnance applications (ref 1) because of its ease of production, high hardness, high melting point, low coefficient of friction, resistance to aggressive chemical environment, and resistance to wear and erosion. High contraction (HC) chromium—also known as “bright chromium”—shrinks after heating and subsequent cooling. It is often used to reduce galling, friction, wear, and erosion and is the material of choice for production refractory coatings, which protect the cylinder bore of gun tubes against high temperature and high pressure-induced deterioration. However, HC chromium is low in tensile strength and contains characteristic cracks. These cracks allow combustion gases to penetrate and corrode the chromium coating and steel substrate—thereby reducing lifetime and causing failure.

The softer and almost crack-free low contraction (LC) chromium exhibits less contraction after heating (refs 2, 3), because it contains less impurities than HC chromium. LC chromium has been considered as a promising alternative to HC chromium for protecting cylinder bores against wear and erosion, and several investigators continue research and development in HC and LC electrolytic chromium deposition. For example, anisotropy and residual stress in HC chromium specimens were deduced using a Matlab matrix inversion method (refs 4, 5). Chen *et al.* studied improved LC chromium electrodeposition (refs 6, 7) and laminated HC-LC chromium electrodeposition (ref 8), while Pan *et al.* studied the optimization of the LC plating parameters (ref 9). Acoustic emission during chromium plating (ref 10) and the effects of pulse plating LC chromium (ref 11) have also been investigated. All of this research provides the crystallographic texture basis for the germination and growth of electrodeposited HC and LC chromium using an enhanced, high-resolution, three-dimensional pole figure technique. Hardness, topography, and microstructure analyses were also performed.

## EXPERIMENTAL PROCEDURE

### Immersion and Flow-Through Chromium Plating Facilities

The manufacturing processes for production chromium plating have been summarized by Collins (ref 12). Both production and laboratory chromium plating systems use an immersion plating technique, although flow-through chromium plating facilities for chromium deposition are being constructed. The immersion plating solution was prepared by dissolving 256 gram/liter of chromic acid ( $\text{CrO}_3$ ) and 2.56 gram/liter of sulfuric acid ( $\text{H}_2\text{SO}_4$ ) into distilled water. HC chromium was plated at low temperature and low current density; LC chromium was plated at high temperature and high current density. Table 1 gives the experimental conditions for the five specimens under investigation—a production HC chromium specimen deposited on steel; a production LC chromium specimen deposited on steel; and three laboratory LC specimens deposited on copper with and without pulse current plating and cathode anode rotation. All of the LC chromium specimens were deposited using the same temperature and current density. In the pulse plating and cathode-anode rotation analysis, the specimen acted as the cathode, and the wire mesh platinum titanium acted as the anode. Pulse plating used 1.0 ms on-time and 1.0 ms off-time. The thickness measurements in Table 1 were made with a Leitz metallographic microscope.

**Table 1. HC-LC Specimen Plating Conditions and Measured Thickness**

<b>Specimens</b>	<b>Production Condition</b>	<b>Substrate</b>	<b>Thickness</b>
HC CR (s213)	Production immersion system, 55°C	Martensite Steel	122 $\mu$ (4.8 mil)
LC CR (lccrc1)	Production immersion system, 85°C	Martensite Steel	130 $\mu$ (5.1 mil)
LC A	Laboratory immersion system, 85°C, 10 amps, rotated at 100 rpm	Cu plate	56 $\mu$ (2.2 mil)
LC B	Laboratory immersion system, 85°C, 10 amps, pulse plating	Cu plate	25 $\mu$ (1.0 mil)
LC C	Laboratory immersion system, 85°C, no rotation, no pulse plating	Cu plate	63 $\mu$ (2.5 mil)

#### Experimental Technique and Enhanced High-Resolution Pole Figure Analysis

An x-ray diffraction study was performed on a Scintag 2000 four-axis diffractometer using Cu radiation. Conventional Bragg two theta measurements provided qualitative information about the preferred orientation in polycrystalline materials. Texture measurement using the pole figure technique for each orientation mapped a stereographic projection of the crystalline plane normal and provided a statistical distribution measurement of grain orientations. The conventional x-ray pole figure technique using the ( $\theta$ - $2\theta$ ) geometry limited “chi” and “phi” analysis to 5° steps for convenient orientation distribution function (ODF) evaluation and did not allow an analysis of the finer steps. When the texture was tight—such as in studies of epitaxial semi-conductor thin films—large steps caused distorted or even missed peaks. The enhanced, high-resolution, three-dimensional pole figure technique that had no limit on step size and a Z-axis representing intensity was developed to facilitate studies of grain orientations (ref 13). A pole figure generated with a chi range of 0° to 80° and a phi range from 0° to 360° contained 2K information when using 5° steps—compared to 58K information when using 1° steps.

#### Texture Analysis in HC and LC Electrolytic Chromium Deposition

Figure 1 compares the diffraction patterns of HC CR and LC CR specimens and 140 mesh chromium powder using Cu K $\alpha$  radiation. The top figure gives the result for LC chromium on steel with superimposed International Center for Data Diffraction (ICDD) database for chromium. It also shows the near random crystalline orientation of LC chromium on steel. The

middle figure shows the results for HC chromium on steel, which has a 98% preferred (222) orientation. For comparison purposes, the bottom figure gives the diffraction pattern for a 140 mesh chromium powder specimen.

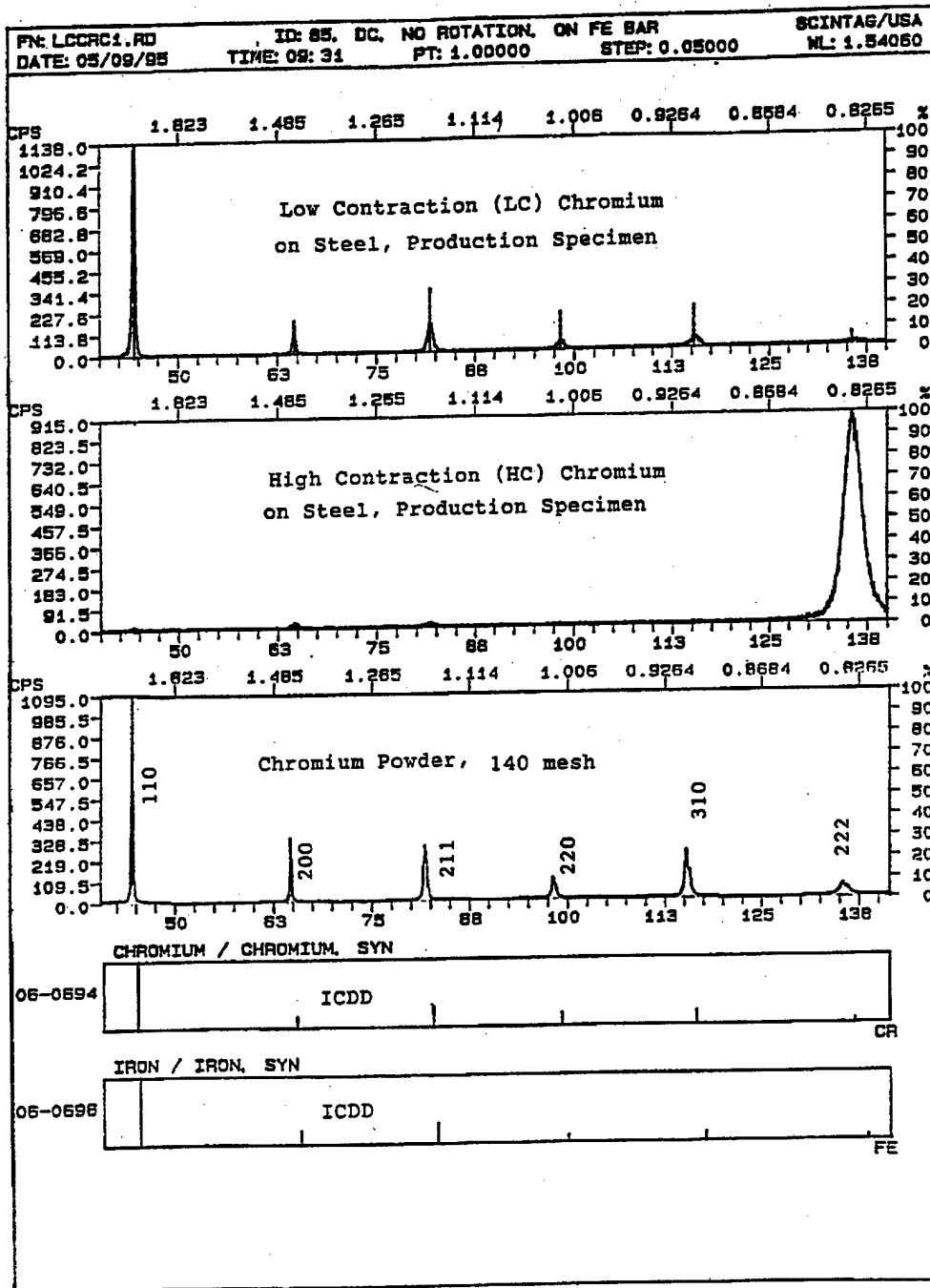


Figure 1. X-ray diffraction patterns of HC and LC chromium deposits on steel substrate compared to 140 mesh chromium powder

In Figure 2, texture in production HC chromium is compared to production LC chromium using the (200), (211), and (220) pole figures. In the upper figures for HC chromium on steel, the following was observed—(200) pole ring structure at  $\chi = 54.7^\circ$ ; (211) pole ring structure at  $\chi = 19.5^\circ$  and  $\chi = 61.9^\circ$ ; and (220) pole ring structure at  $\chi = 35.3^\circ$ . Chromium is a bcc crystal, and, from the intensity and location of the ring structures, we concluded that HC chromium contains highly oriented  $\langle 111 \rangle$  fiber texture. All crystalline planes were preferentially oriented—with the  $\{111\}$  planes parallel to the specimen surface with perfect azimuth symmetry around the fiber axis. In the lower figures for LC chromium, pole figures for (200), (211), and (220) reflections showed near random crystallite distribution. In Figure 3, enhanced (211), (110), and (111) pole figures are shown for highly textured HC chromium.

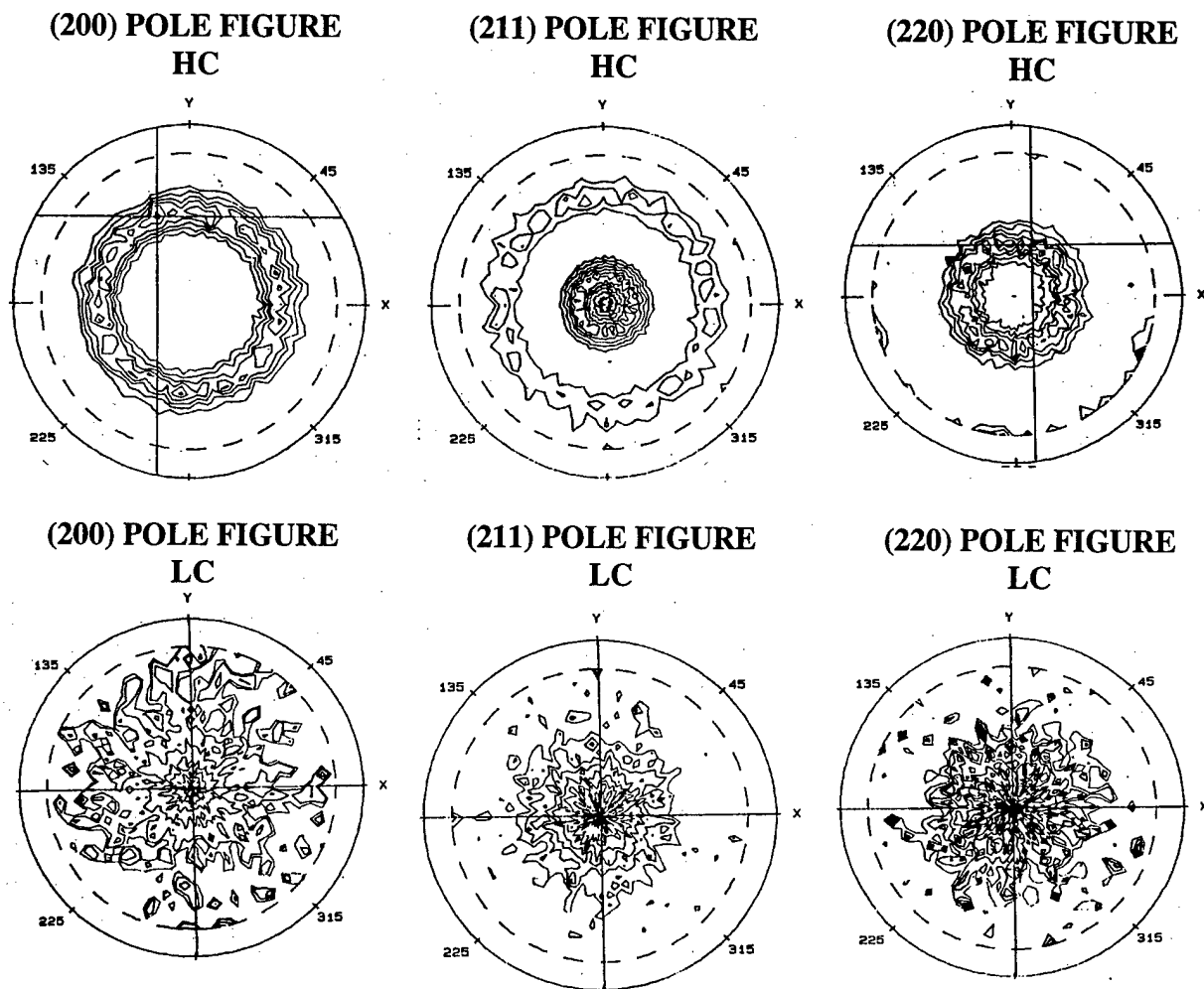


Figure 2. Grain orientation in HC and LC chromium deposits on steel substrate using Cu  $K\alpha$  radiation

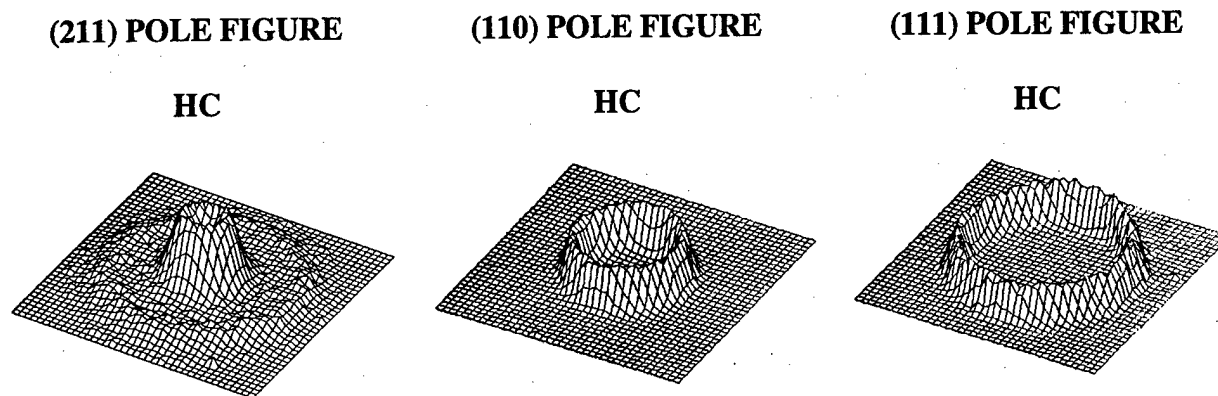


Figure 3. High-resolution analysis and three-dimensional display of pole figures for textured chromium

#### Laboratory Cathode-Anode Rotation and Pulse Current Plating Experiments

Figure 4 gives the diffraction scans for three laboratory LC chromium specimens—plated on copper substrate at 85°C and using the same high current density as in production LC chromium—using copper radiation. From top to bottom, the three figures represent specimens with cathode-anode rotation, with pulse plating, and with no rotation and no pulse plating. The relative percentage intensity for each reflection was computed from an iterative profile fitting of the area under the diffraction peak according to a Pearson VII diffraction peak profile and using raw peak height. Preferred (211) and (222) orientations were observed in all three specimens—with only small crystallographic differences noted. The preferred (211) orientation and the (222) orientation for specimens plated at 85° was no surprise because it had also been observed in laboratory LC chromium specimens on copper substrate at various current densities (ref 9). Its appearance in specimens LC A, LC B, and LC C—but not in specimen LC CR, which was plated under the same temperature and current density—can be attributed to the difference in substrates and to the controlled production process.

#### Hardness, Microstructure, and Other Physical Properties

Because the observed textures in production HC and LC chromium are drastically different, their physical properties are radically different as well. Hardness in electrolytic chromium can be caused by crystalline texture, hydrogen and oxygen content, internal stress, or grain size (ref 6). Because crystalline anisotropy determines the elastic-plastic properties in textured materials (such as Young's modulus, Poisson's ratio, and yielding), hardness in HC and LC chromium is expected to be very different. Table 2 provides hardness measurements, which were taken using a Knoop diamond indenter at a 50g load. Each data point is an average of five measurements. HC chromium plated at low temperature and low current density has a hardness of approximately 1000 Knoop, while LC specimens plated at high temperature and high current density have a hardness of approximately 600 Knoop—independent of the substrate.

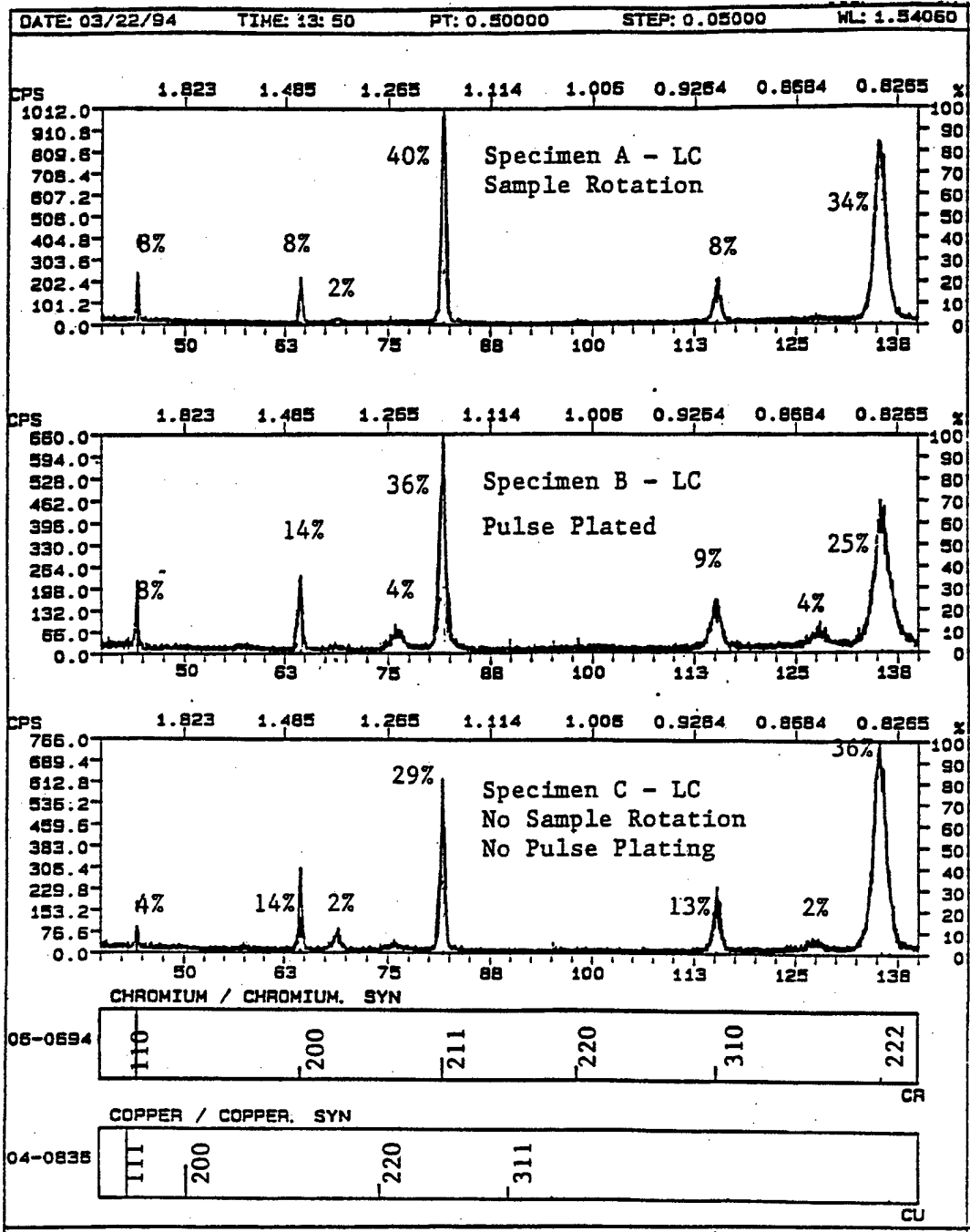


Figure 4. X-ray diffraction patterns for LC chromium plated onto Cu substrate using cathode-anode rotation and pulse current plating LC A, LC B, and LC C

A scanning electron microscope and a Leitz MM5 metallography camera were used to examine the topography and cross-sectional microstructure of the deposits. In Figure 5, the topography and microstructure of production HC chromium is compared to production LC chromium. The top left figure shows an HC chromium microstructure with extensive vertical cracks, and the top right figure shows an LC chromium microstructure with much reduced crack density because LC chromium is considered to be almost crack-free. The bottom left figure shows HC chromium topography, which has extensive surface cracks, and the bottom right figure shows LC chromium surface topography. The extensive cracks in the HC chromium can be attributed to high tensile residual stress during deposition.

Table 2 lists other HC and LC chromium physical properties that can be affected by the differences in crystalline texture. These properties include fracture strength, density, thermal behavior upon heating, and wear and erosion performance. Figure 6 gives the thermal expansion coefficient of production HC and LC chromium and shows the HC and LC nature of the coatings (ref 8). Figure 7 compares the wear and erosion performance of the LC coatings to the HC coatings (ref 6). In the plot, LC chromium on steel shows better wearing characteristics than HC chromium on steel.

**Table 2. Characteristics of Electrolytic HC and LC Chromium Depositions**

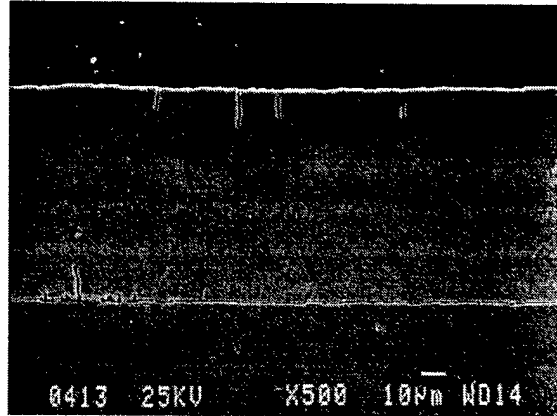
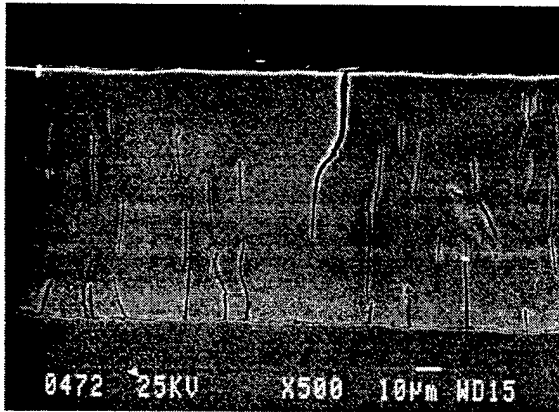
Properties	Production HC	Production LC	Laboratory LC A	Laboratory LC B	Laboratory LC C
Physical Appearance	Shiny	Dull	Dull	Dull	Dull
Preferred Orientation	(111) fiber	Near random	(211) (111)	(211) (111)	(211) (111)
Knoop Hardness	1005+/-52.9	600.2+/-20.3	527.1+/-22.8	692.9+/-27.9	590.8+/-22.9
Cracks	Yes	No	No	No	No
Ductility	Brittle	More ductile			
Fracture Strength	105 MPa	343 MPa			
Deposition Rate	25.4 $\mu$ /hr (1 mil/hr)	76.1 $\mu$ /hr (3 mil/hr)			
Thermal Contraction	Shrank after heating	Negligible contraction			
Density (gram/cm <sup>3</sup> )	7.01	7.16			

**HC CR**

**LC CR**

**HC MICROSTRUCTURE**

**LC MICROSTRUCTURE**



**HC SURFACE TOPOGRAPHY**

**LC SURFACE TOPOGRAPHY**

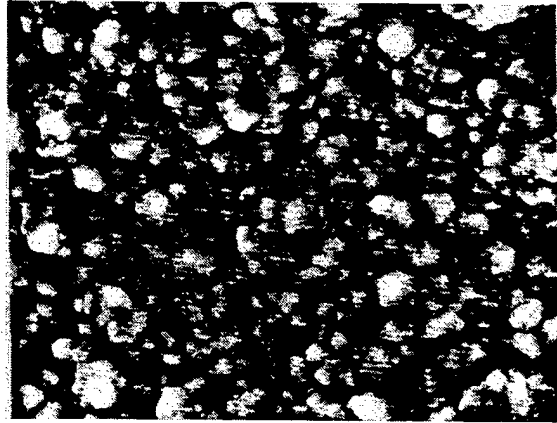
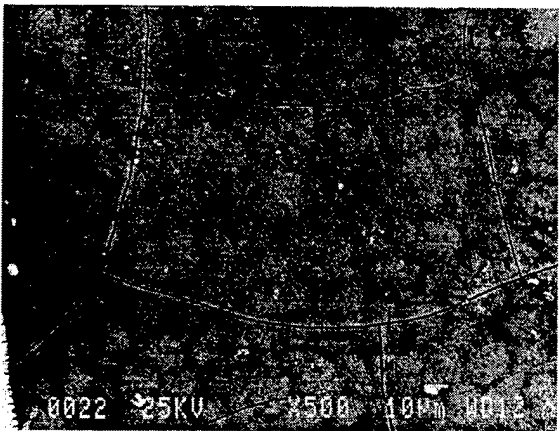


Figure 5. HC and LC chromium electron microscope topography and microstructure

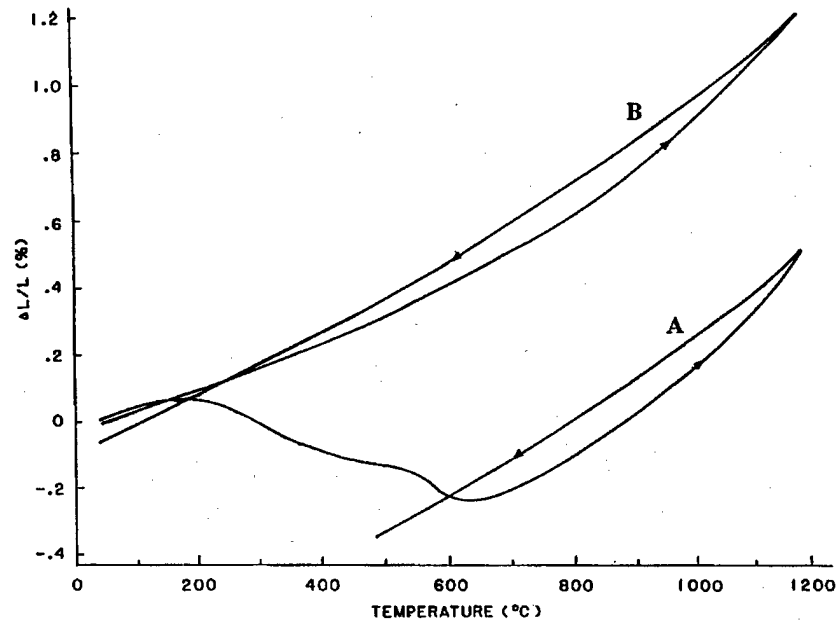


Figure 6. Thermal expansion coefficient in HC-LC chromium deposition from reference 8 (Curve A - HC chromium, Curve B - LC chromium)

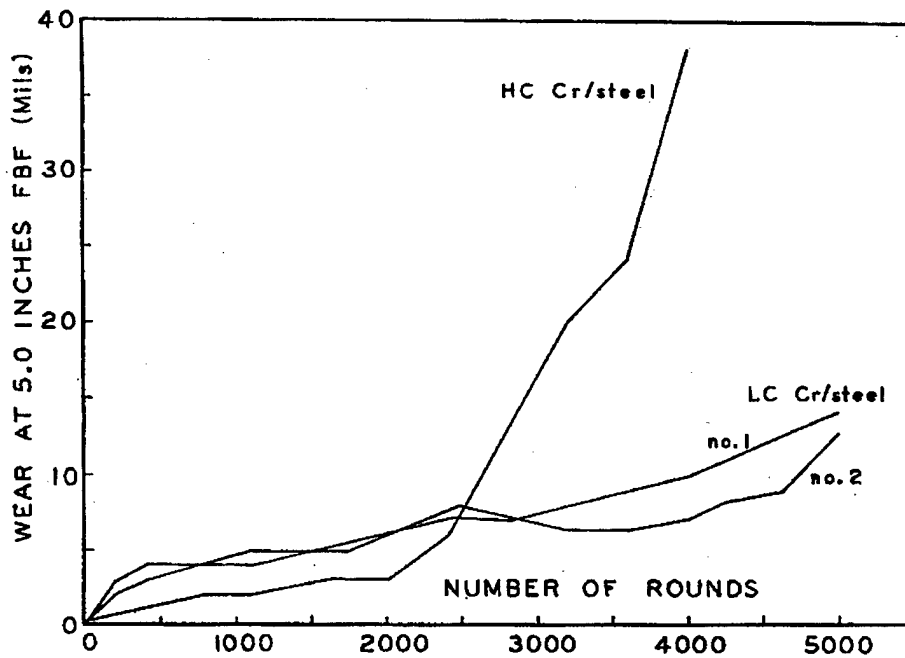


Figure 7. Wear and erosion performance in HC-LC chromium on steel from reference 6

## CONCLUSIONS

Because crystalline grain orientation plays an important role in controlling the physical properties of materials, engineered crystalline texture has often been used to optimize material behavior for specific applications. Changing the deposition parameters in electrolytic chromium deposition—such as temperature and current density—drastically changes the crystalline texture. For wear and erosion applications, randomly-oriented LC chromium is superior to highly textured HC chromium. Other conclusions are as follows.

1. Production HC chromium plated on steel at a lower temperature and lower current density exhibited predominately  $\langle 111 \rangle$  fiber texture.
2. LC chromium plated on steel at a higher temperature and higher current density exhibited an almost random texture.
3. Laboratory LC chromium coatings plated on copper with or without sample rotation and pulsed current plating exhibited (111) and (211) preferred orientations.
4. Plating bath temperature and current density are the most critical factors affecting grain orientations. Relative rotation of the specimen and pulse current plating conditions play a less important role.
5. The drastic transition from strong fiber-textured HC chromium to random-oriented LC chromium is accompanied by marked improvement in crack density, microstructure, hardness, deposition, thermal behavior upon heating and cooling, and wear and erosion performance.

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