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TECHNICAL REPORT ARCCB-TR-89024

**OPTIMIZATION OF PLATING
PARAMETERS FOR LOW CONTRACTION
CHROMIUM ELECTRODEPOSITS**

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Electrodeposition of low contraction (LC) chromium has been investigated in order to deposit a high tensile strength and crack-free chromium coating. The plating parameters--current density, chromic acid/sulfuric acid ratio, and chromium(III) concentration--were varied and a systematic study was performed. A chromic acid concentration of 250 g/l and a bath temperature of 85°C were maintained constant throughout the study. The deposit is crack-free having a typical columnar microstructure of fibrous grains with a <211> preferred (CONT'D ON REVERSE)		

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INTRODUCTION

The electrodeposition of chromium has been in existence for more than 150 years (refs 1,2). Chromium is electrodeposited for many reasons including its decorative finish, high hardness, and corrosion resistance properties. The U.S. Army electroplates chromium on the bore of gun tubes for protection against wear and erosion. High contraction (HC) chromium is the method of electrodeposition used by the Army. Although desirably hard and often used for its bright deposit, HC chromium is low in tensile strength, highly stressed, and contains many microcracks. When a projectile is fired inside a gun tube, high temperature combustion gases under extreme pressure are produced at the bore surface. Gases such as carbon monoxide, carbon dioxide, hydrogen, nitrogen, and water penetrate the microcracks in the HC electrodeposits and corrode the chromium, while at the same time exposing the base metal (steel).

A virtually crack-free chromium deposit called low contraction (LC) chromium (refs 3,4) was produced that is softer and stronger in tensile strength than HC chromium. In addition, LC chromium provides improved corrosion protection--erosion resistance. In the absence of microcracks, the propellant gases cannot penetrate the deposit surface and interact with the gun steel. It is apparent that LC chromium, being stronger and crack-free, is more suitable than HC chromium for protecting gun tubes.

Previous reports by Chen et al. (refs 5,6) and Cullinan et al. (ref 7) dealt with the development of the LC chromium plating process, but neither recommended optimal plating conditions. This study systematically investigates

References are listed at the end of this report.

the LC chromium plating process to determine the optimal plating conditions needed to obtain increased hardness and tensile strength. An increase in hardness and tensile strength should result in an increased firing life for the gun tubes. Standard LC chromium is plated at 85°C, however the optimum current density (CD), chromic acid/sulfuric acid ($\text{CrO}_3/\text{H}_2\text{SO}_4$) ratio, and chromium (Cr)(III) concentrations have yet to be determined. The purpose of this study is to determine these parameters and correlate them with the microstructure, topography, tensile strength, and hardness of the chromium deposit and the optimum cathode current efficiency (CCE) of the plating process.

EXPERIMENTAL PROCEDURE

Plating Solutions

A chromic acid solution was prepared by dissolving 270 g/l of technical grade CrO_3 and 2.3 g/l of H_2SO_4 in distilled water. The solution was pre-electrolyzed for 250 A·hr/l at a CD of 15 A/dm². It was then filtered and adjusted by adding the appropriate amounts of distilled water and H_2SO_4 to form stock solutions containing CrO_3 at 250 g/l and H_2SO_4 at 2.5 g/l.

The plating solutions for $\text{CrO}_3/\text{H}_2\text{SO}_4$ ratios of 90/1, 80/1, 70/1, and 60/1 were prepared by adding the appropriate amounts of H_2SO_4 to one-liter amounts of the stock solution. Plating solutions containing various Cr(III) concentrations were prepared by adding 10, 20, and 30 g/l of Cr(III) into stock solutions, respectively. This was accomplished by reacting the appropriate amounts of oxalic acid solution ($\text{H}_2\text{C}_2\text{O}_4$) to reduce chromium from hexavalent to trivalent. The analysis showed that the stock solution contained 4.0 g/l of Cr(III), therefore the Cr(III) concentrations were 14, 24, and 34 g/l, respectively.

Electrodes

In order for the plated specimens to be used in tensile tests, they were electroformed in the shape of cylinders. A copper tube with 5/32-in. outer diameter (O.D.) was used as the mandrel (cathode) and masked to have a plating area of 10 cm². It was placed vertically in the center of the beaker and rotated at 150 rpm during plating. A flat platinum anode with a surface area of 20 cm² was placed opposite the cathode in one side of the beaker. The distance between the cathode and the anode was 4 cm. For x-ray diffraction (XRD) analysis, the specimens were prepared by depositing LC chromium on a 1.4 by 1.4 cm copper cathode that was masked on one side during plating, and it was positioned and rotated in the same manner as the cylindrical cathode.

Electrodeposition

The plating solution was kept at 85°C and agitated with a Teflon-coated magnetic stirrer. Except when specially mentioned, a standard plating condition with a CD of 120 A/dm², a CrO₃/H₂SO₄ ratio of 100/1, a Cr(III) concentration of 4.0 g/l, and a plating time of 2 hours was used. For the investigation of CD variation, specimens were plated at various plating times to keep the total charge transfer (A·hrs) constant. For the studies on CrO₃/H₂SO₄ ratio and Cr(III) concentration, the solutions mentioned in the previous section were used, while all other parameters in the standard plating condition were kept constant.

Microstructure

A scanning electron microscope (SEM) was used to examine both the topography and the cross-sectional microstructure of the deposits. The cross-sectional microstructure was also examined using a Leitz MM5 metallograph. The

crystal structure of the deposits was studied using x-ray diffraction. A Phillips Norelco diffractometer with Mo $K\alpha_1$ radiation was used.

Mechanical Properties

A tensile test was performed with a table model Instron machine to measure the ultimate tensile strength (UTS) of the deposited cylindrical specimen. The test procedures were the same as those described in previous reports (refs 5,6). In order to reduce hydrogen embrittlement of chromium, the copper mandrel in the tensile specimen was dissolved in a cooled (0°C), diluted (50 percent) nitric acid solution (HNO_3).

The hardness measurements were made with a Knoop microhardness indenter using a 50 g load.

RESULTS AND DISCUSSION

Six specimens were prepared for each of the plating conditions investigated. The results, averaged from the specimens in terms of CCE, microhardness, and UTS measurements, are summarized in Table I.

Effects of Current Density

In LC chromium plating, the CD has a pronounced effect on the current efficiency, microstructure, and mechanical properties. Figure 1 shows the CCE, UTS, and hardness as functions of the CD. The CCE increased from 8.1 to 12.9 percent as the CD was increased from 60 A/dm² to 120 A/dm². This agrees well with Kasaaian's high current efficiency theory (ref 8), i.e.,

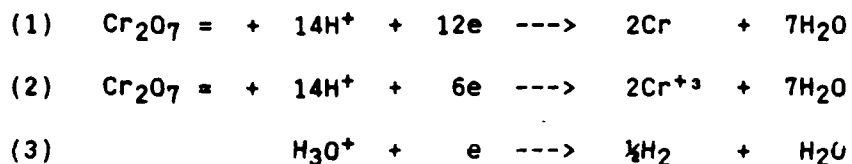


TABLE I. SUMMARY OF LC CHROMIUM* ELECTRODEPOSITION

Specimen Group No.	CrO ₃ /H ₂ SO ₄ Ratio	Cr(III) (g/l)	CD (A/dm ²)	CCE (%)	Hardness KHN	UTS (ksi)
RAT-1	60	4.0	120	15.2	587	71-79
RAT-2	70	4.0	120	14.3	633	74-83
RAT-3	80	4.0	120	13.8	689	75-87
RAT-4	90	4.0	120	13.6	711	76-87
RAT-5	100	4.0	120	12.9	760	76-87
CR3-1	100	4.0	120	12.9	760	73-80
CR3-2	100	14.0	120	13.1	671	58-67
CR3-3	100	24.0	120	13.2	626	53-61
CR3-4	100	34.0	120	13.3	592	53-62
CCD-1	100	4.0	60	8.1	546	25-40
CCD-2	100	4.0	70	10.3	537	29-52
CCD-3	100	4.0	80	10.9	544	43-67
CCD-4	100	4.0	90	11.6	606	68-85
CCD-5	100	4.0	100	12.3	663	78-88
CCD-6	100	4.0	120	12.9	760	61-77

*Chromic acid solution 250 g/l, bath temperature 85°C.

There is a greater tendency for reaction (1) to take place than the other reactions as the CD is increased. Hence, a greater percentage of the current goes toward depositing chromium.

The hardness did not vary significantly with a change in low CD (from 60 to 80 A/dm²), but it increased linearly from 540 KHN at 80 A/dm² to 760 KHN at 120 A/dm².

As a result of high internal stress, growth defects in the chromium, and premature failure in the tensile test, the UTS data were scattered (shaded area, Figure 1). The UTS values increased from a range of 20 to 40 ksi at 60 A/dm² to a range of 78 to 90 ksi (a maximum) at 97 A/dm². The UTS decreased when the CD was increased beyond 97 A/dm². As shown in Figure 1, the UTS follows the same slope as the hardness. The two lines appear very similar until the UTS value reaches the CD value of 97 A/dm², however, beyond that the internal stresses become too great, cracking results, and the UTS values decrease.

The topographical photomicrographs of LC chromium plated at different CD's are shown in Figure 2. The grain size of the chromium deposits increases as the CD decreases. Higher tensile strength and hardness are often a result of larger grain size because growth defects and high stress risers, which result during coalescence of crystallites, occur more frequently in smaller grains.

Figure 3 includes SEM photomicrographs showing the cross-sectional microstructure of an LC chromium sample deposited at 120 A/dm². This columnar fibrous structure shown is typical of most of the deposits. The LC chromium deposits were crack-free and 100 percent dense (density measurements were 7.26 g/cm³). Figure 4 shows the microstructure of LC chromium deposited at 100 A/dm². The photomicrographs of the cross section show a strong cross-fibrous structure. This may account for the high tensile strength at 97 A/dm².

A series of x-ray diffraction patterns of LC chromium electrodeposited at various CD's is shown in Figure 5. A strong 211 peak intensity appears on each of the patterns indicating that the deposits have a $\langle 211 \rangle$ preferred orientation. In comparing the intensities of the 211 peaks in all four patterns, it is further noted that the higher the CD used for plating a sample, the stronger the $\langle 211 \rangle$ preferred orientation. This is also consistent with the decreasing 110 peak intensities with the increasing CD's, since the 110 peak is strong when the deposit is random (no preferred) orientation.

Effects of Sulfuric Acid

The effects of H_2SO_4 on the electrodeposition of chromium are illustrated in Figure 6. The CCE decreased from 15.2 to 12.9 percent as the CrO_3/H_2SO_4 ratio increased from 60/1 to 100/1 and the hardness increased from 590 to 760 KHN for the same increase in acid ratio. As the CCE decreased, the amount of hydrogen liberated at the cathode increased, and the possibility of hydrogen embrittlement increased, resulting in a more brittle and harder chromium.

The UTS increased slightly as the CrO_3/H_2SO_4 ratio increased. This rise was not as pronounced as the UTS increase when the CD was increased. Once again, the UTS reached a maximum and then gradually decreased. This may be the result of increased internal stresses which were responsible for the onset of microcracks becoming worse as the CrO_3/H_2SO_4 ratio increased beyond 90/1.

In examining the cross section of the deposits with a scanning electron microscope, it is seen that the microstructures of the 60/1 and 70/1 acid ratios have slightly coarser grains than the 80/1, 90/1, and 100/1 ratios. Accordingly, the tensile strengths obtained from these samples were lower for the 60/1 and 70/1 ratios than for the 80/1, 90/1, and 100/1 ratios.

The H_2SO_4 content in the LC chromium plating solution has an effect on the crystal structure of the deposit. Figure 7 illustrates the x-ray diffraction patterns of chromium deposits which were plated from solutions with various CrO_3/H_2SO_4 ratios. The patterns clearly demonstrate that a strong $\langle 211 \rangle$ orientation is gradually converted to a strong $\langle 100 \rangle$ preferred orientation when the amount of H_2SO_4 is increased (acid ratio decreased).

Effects of Chromium(III) Concentration

Figure 8 shows CCE, UTS, and hardness as functions of the Cr(III) concentration. It is shown that increasing the Cr(III) concentration from 4.0 to 34.0 g/l increases the CCE from 12.9 to 13.3 percent. This correlation agrees well with Kasaaian's data (ref 8). It is believed that the cathode film contains a significantly higher concentration of Cr(III) than the plating solution. A portion of the current must be used to produce enough Cr(III) to compensate the Cr(III) loss by diffusion and to maintain a stable cathodic film. As more Cr(III) is added to the plating solution, the diffusion rate is reduced. As a result, less Cr(III) has to be produced and a greater portion of the current goes toward depositing chromium.

Although increasing the Cr(III) concentration has a positive effect on the CCE, its effect on such mechanical properties as hardness and UTS is detrimental. The hardness decreased from 760 to 590 KHN as the Cr(III) concentration was increased from 4.0 to 34.0 g/l. In the same manner, the UTS values decreased from a range of 73 to 80 to a range of 53 to 62 ksi for the same Cr(III) increase in concentration.

In examining the topography of the LC chromium deposits at different Cr(III) concentrations, no significant difference was observed. Similarly, the

cross-sectional microstructures of the specimens appear identical to those shown in Figure 3.

Figure 9 shows the x-ray diffraction patterns of LC chromium deposited from the solutions containing various amounts of Cr(III). These patterns show a $\langle 211 \rangle$ preferred orientation which becomes weaker as the Cr(III) concentration is increased.

CONCLUSIONS

The electrodeposition process of LC chromium has been improved by investigating various plating parameters, such as current density, $\text{CrO}_3/\text{H}_2\text{SO}_4$ ratio, and Cr(III) concentration. Based on the results of our experimental studies, the following conclusions can be drawn:

1. Chromium electrodeposited from 250 g/l CrO_3 solution at 85°C is crack-free and has a columnar fibrous grain structure.
2. The cathode current efficiency of deposits made at 250 g/l will be in the range of 8.1 to 15.2 percent depending upon the plating condition.
3. The coating has a strong preferred orientation and the crystallographic planes $\{211\}$ are preferentially oriented parallel to the surface of the coating.
4. On the basis of mechanical properties of the deposits, the following optimum plating conditions were found: a current density of 97 A/dm², a $\text{CrO}_3/\text{H}_2\text{SO}_4$ ratio of 100/1, and a Cr(III) concentration of 4.0 g/l.
5. Under the optimum plating conditions, a tensile strength of 87,000 psi or greater and a maximum hardness of 760 KHN can be found.

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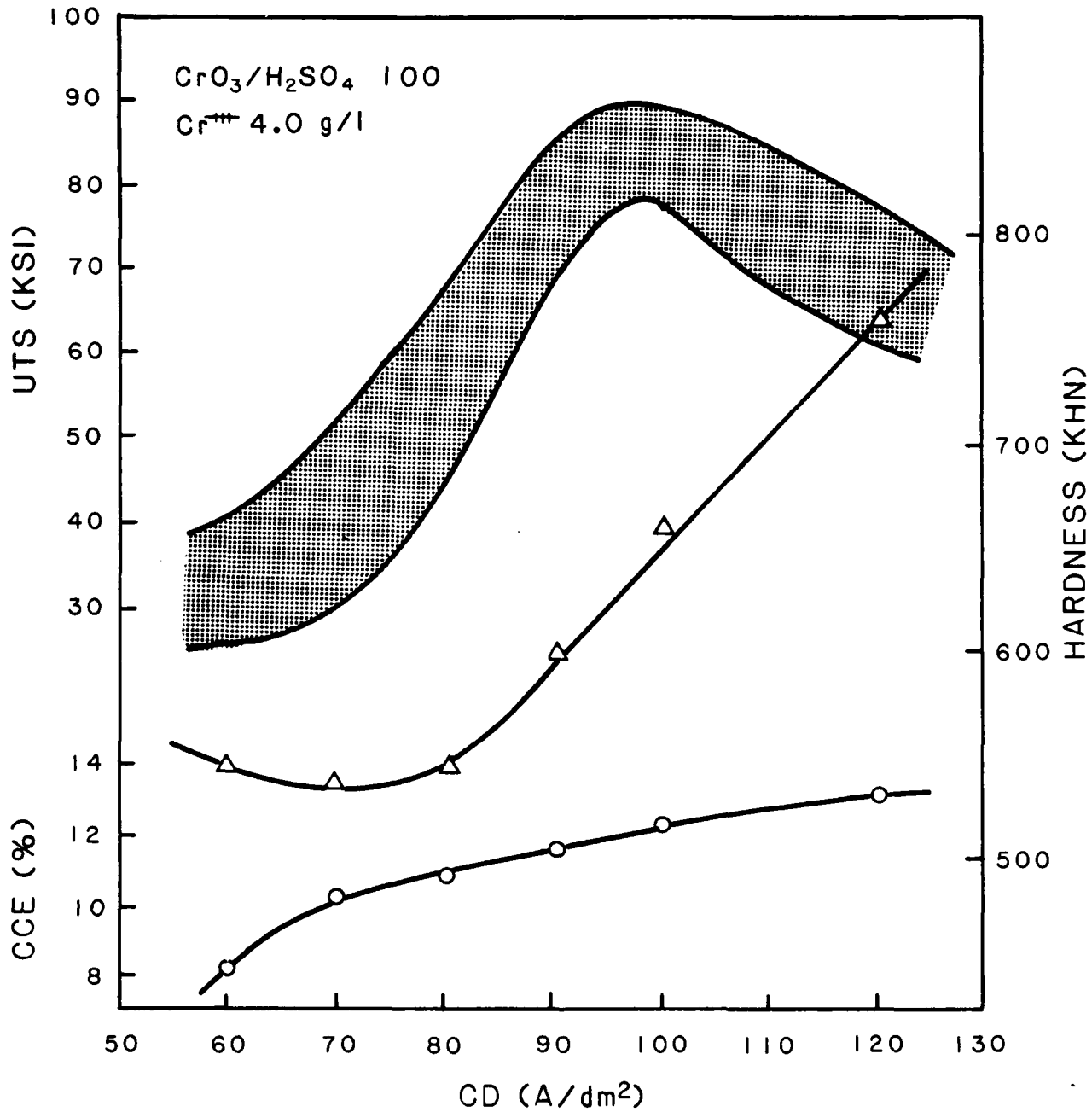
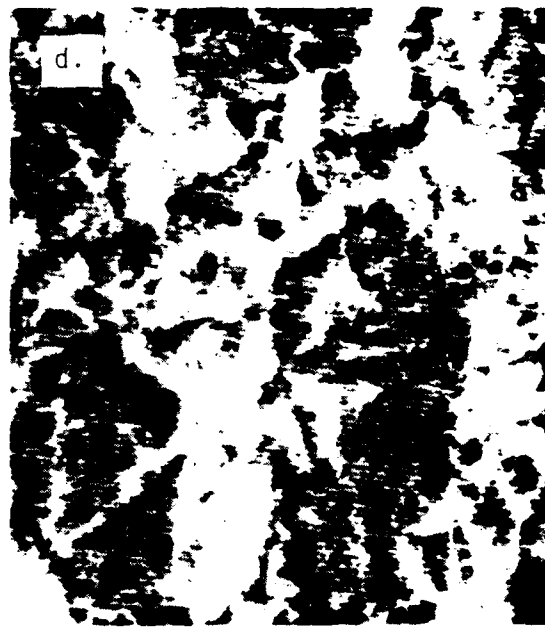
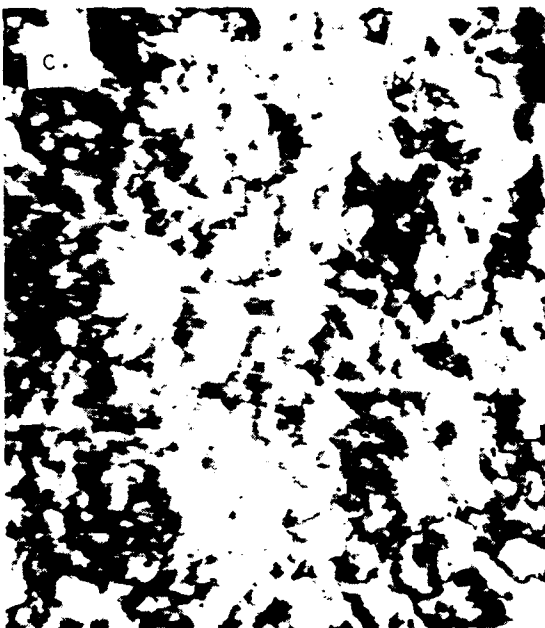
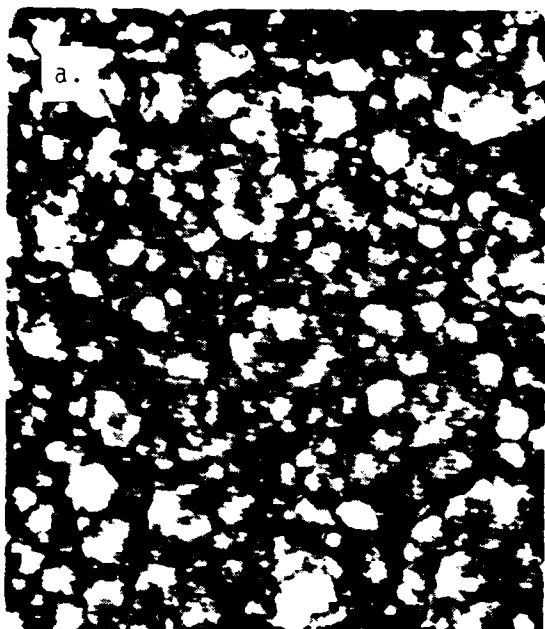


Figure 1. Effects of current density on ultimate tensile strength, hardness, and cathode current efficiency of electrodeposited LC chromium.



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Figure 2. Photomicrographs of topographies of LC chromium electrodeposited at current densities of: (a) 100 A/dm^2 , (b) 80 A/dm^2 , (c) 70 A/dm^2 , and (d) 60 A/dm^2 .

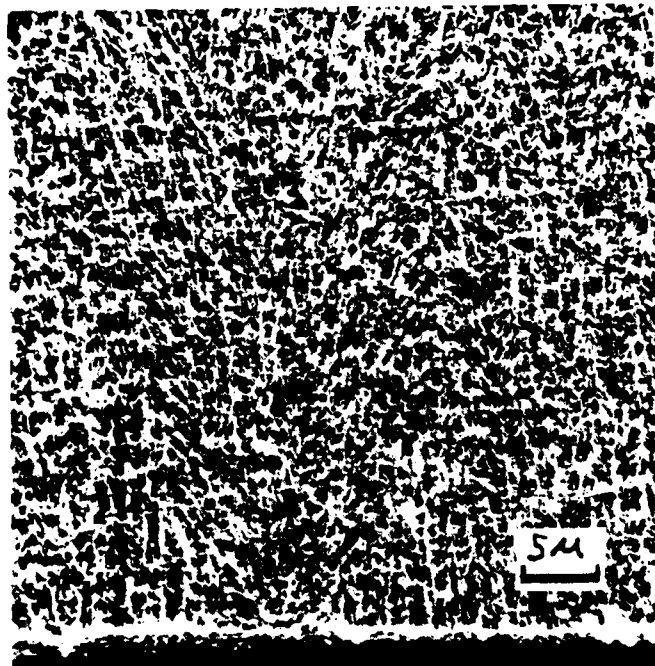
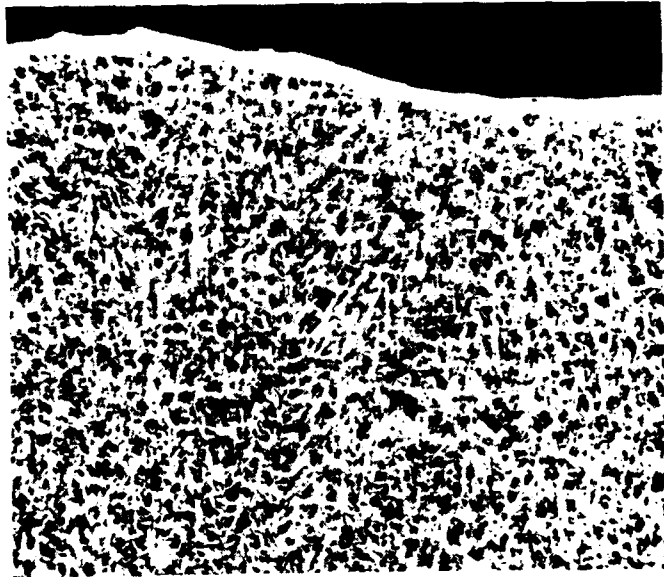


Figure 3. Photomicrographs of electrodeposited LC chromium (near the top and interface of the cross section) showing the typical microstructure of fibrous columnar grains.



Figure 4. Photomicrographs of a cross section of LC chromium electrodeposited at 100 A/dm^2 showing the microstructure with cross-fiber grains.

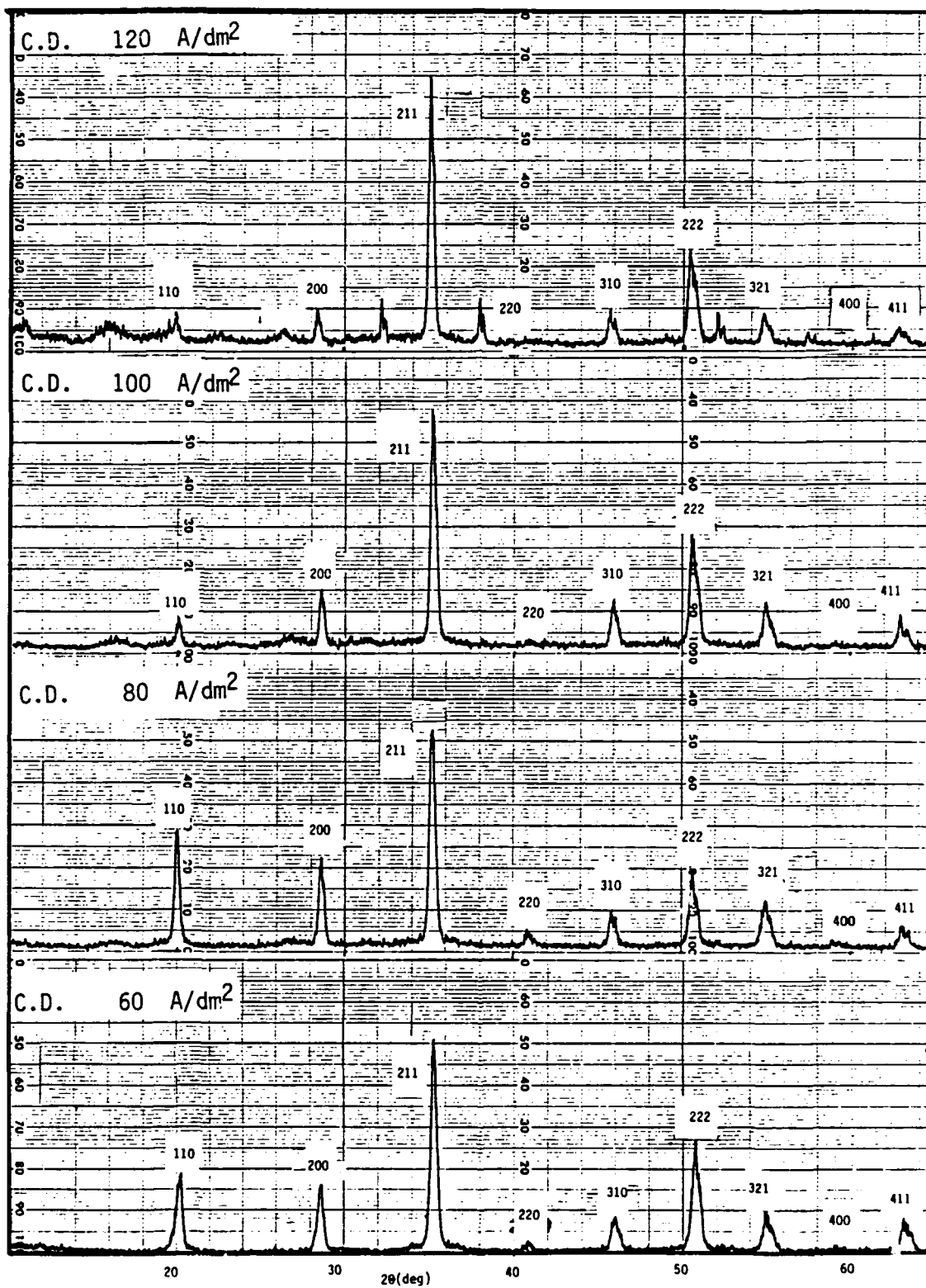


Figure 5. X-ray diffraction patterns of LC chromium electrodeposited at current densities of 120 A/dm², 100 A/dm², 80 A/dm², and 60 A/dm².

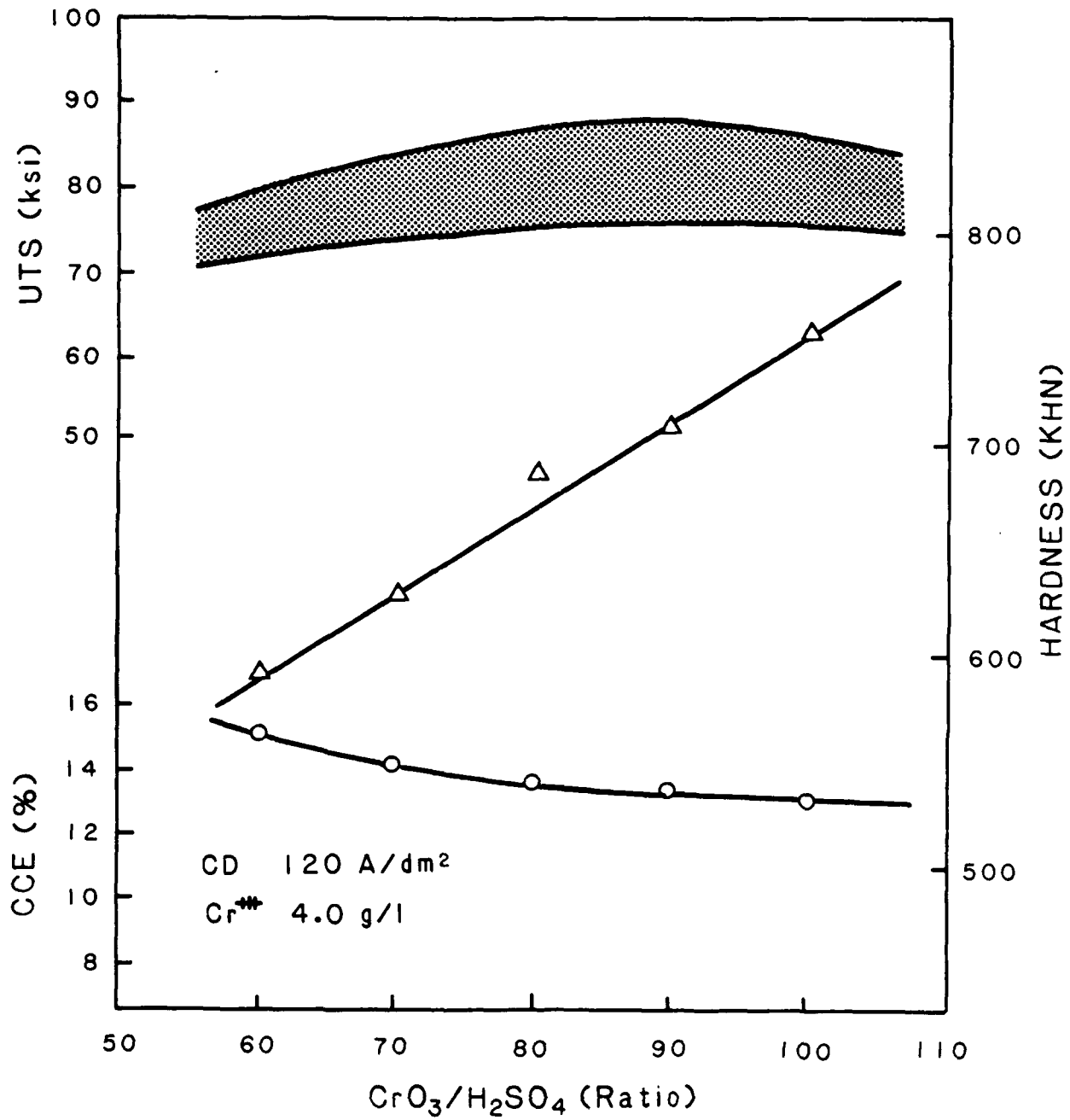


Figure 6. Effects of H_2SO_4 concentration on ultimate tensile strength, hardness, and cathode current efficiency of electrodeposited LC chromium.

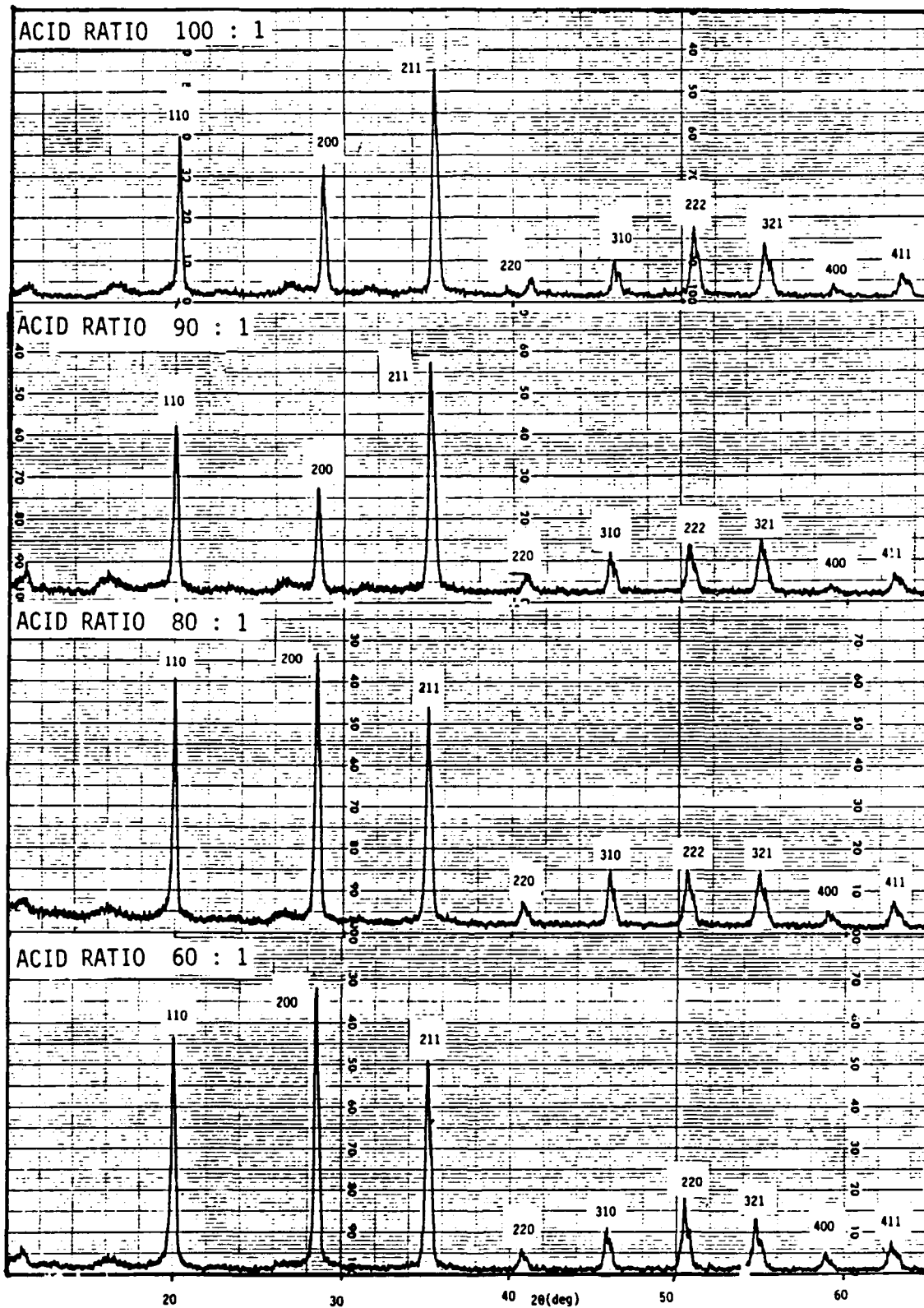


Figure 7. X-ray diffraction patterns of LC chromium electrodeposited from CrO_3 containing various amounts of H_2SO_4 . (Acid ratio of $\text{CrO}_3/\text{H}_2\text{SO}_4$ at 100/1, 90/1, 80/1, and 60/1.)

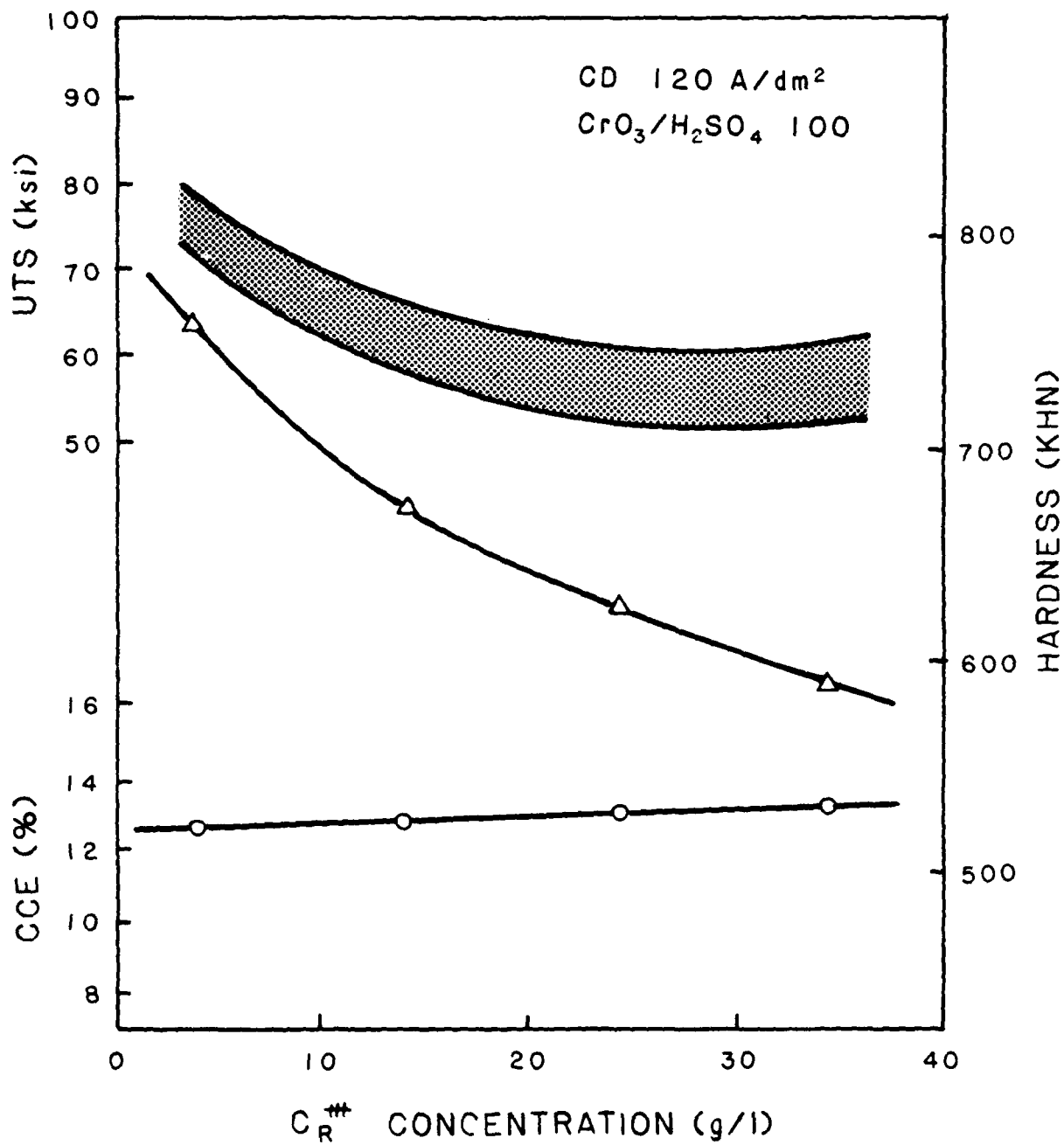


Figure 8. Effects of Cr(III) concentration on ultimate tensile strength, hardness, and cathode current efficiency of electrodeposited LC chromium.

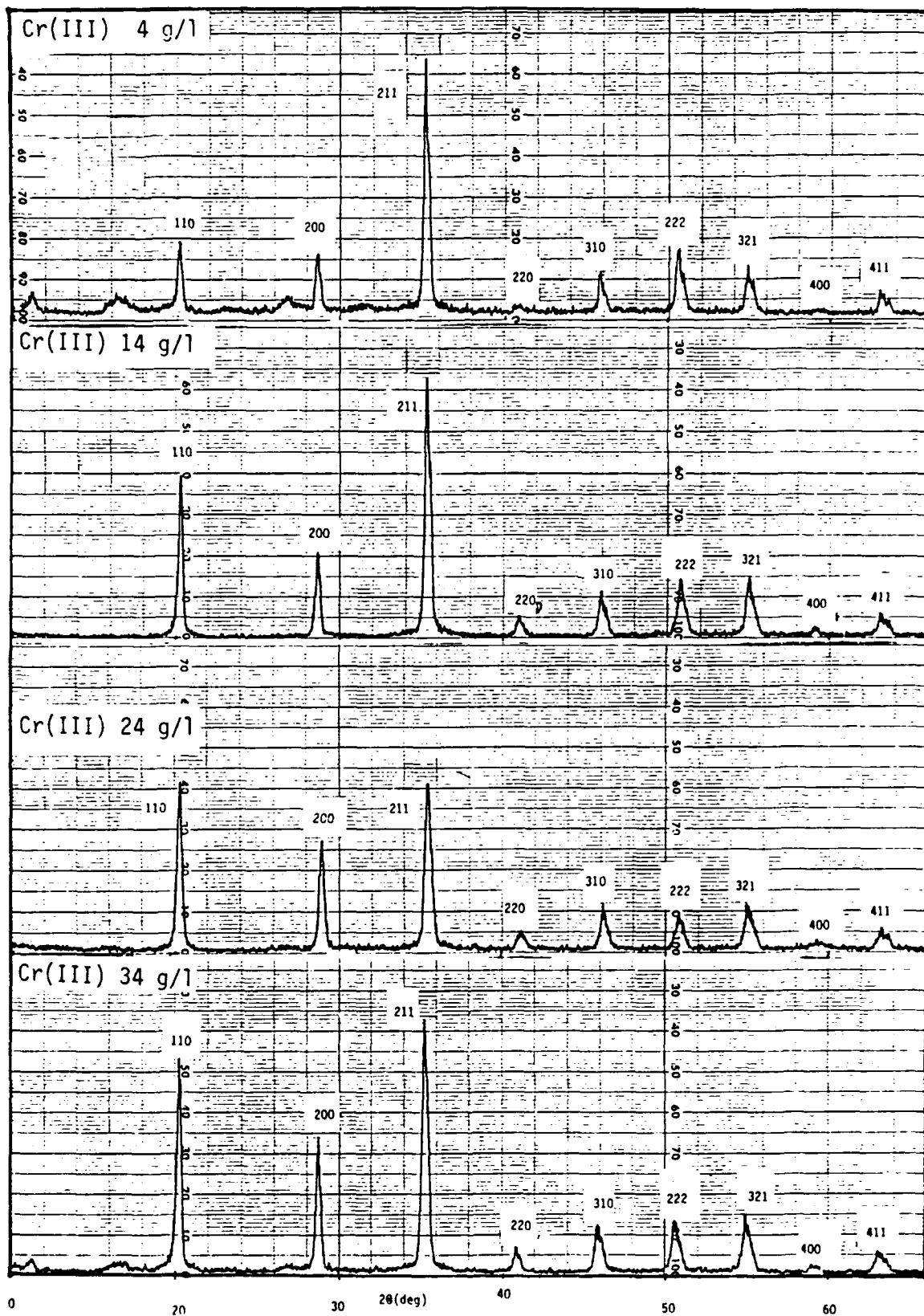


Figure 9. X-ray diffraction patterns of LC chromium electrodeposited from CrO_3 containing Cr(III) at 4 g/l, 14 g/l, 24 g/l, and 34 g/l, respectively.

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