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TECHNICAL REPORT ARLCB-TR-83033

CHARACTERIZATION OF CHYSTALLOGRAPHIC STRUCTURE AND INTERNAL STRESS OF CHROMIUM COATINGS PLATED UNDER CURRENT INTERRUPTIONS

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SEPTEMBER 1983

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Current Interruptions	Texture
Microstructure	Stress
Hardness	Microcrack Morphology

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The effect of interrupted current (IC) plating on the visual crack morphology, crystallography, and residual stresses of electrodeposited chromium has been investigated. Varying the process parameters such as the on/off plating cycle and current density resulted in changing the crystallographic fiber texture of the deposit from the conventional <111> orientation to a combination of <211>, <111>, and a small fraction of randomly oriented crystallites. Under

(CONT'D ON REVERSE)

20. ABSTRACT (CONT'D)

these plating conditions, it was also found that (1) lower amounts of chromium hydride (CrH_x) occur in the deposit, (2) a large decrease in the microcrack density of the deposit occurs, and (3) the deposits tend to become compressively stressed.

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INTRODUCTION

For over 30 years High Contraction (HC) chromium has been used to extend the wear and erosion life of gun barrels. Its attractiveness results from its oxidation resistance and retention of hardness at elevated temperatures and its excellent resistance to friction and wear. The acceptance of electrolytically deposited HC chromium as a good protective coating is limited however, by the large tensile stresses and the well-known crack networks which are always present in the deposit. These two limiting factors being dependent upon the deposition conditions, proved to be the target of numerous investigations. The idea of plating conventional HC crack-free chromium has been reported by Saiddington and Hoey[1] and others.[2-4] Based on the observation that cracks do not form in chromium deposits until a critical thickness of about 0.5 μm is reached, they have demonstrated[1] that by means of controlled current interruptions, the microcracks can be effectively reduced or eliminated. Although there is no simple theory to explain the presence of stresses in electrodeposits, a number of comprehensive reviews[5,6] suggest that their origin is due to the crystal lattice defects which are trapped in the deposit during the electrocrystallization process. Likewise, the evidence from these articles and others suggest that the various plating parameters are the critical factors that control the residual stress of the deposit.

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[1]J. C. Saiddington and G. R. Hoey, Plating, 61, p. 923 (1974).
[2]H. Chessin and E. J. Seyb, Jr., Plating, 55, p. 821 (1968).
[3]H. Fry, Trans. Inst. Met. Finishing, 32, p. 107, (1955).
[4]W. Blum, Plating, 48, p. 613, (1961).
[5]R. Weil, Plating, 57, p. 1231 (1970).
[6]J. B. Kushner, Plating, 60, p. 1248 (1973).

On the basis of the above evidence, the current work was undertaken to investigate the crystallography and residual stresses in electrodeposited chromium produced as a function of plating variables, as a means of formulating the direction towards property improvements. In particular, the investigation was aimed at the effect of interrupted current (IC) chromium plating at various current densities on the visual crack morphology, crystallographic structure, and residual stress state of the deposit.

EXPERIMENTAL

A standard plating system was set up in which a Flexopulse periodic current-interruptor made by Rapid Electric was placed in series with the D.C. power supply to allow IC plating using several preselected on/off cycles. All of the IC chromium deposits for this study were prepared using a conventional chromic acid solution which had been aged for 250 A-hr/ℓ and maintained at the composition of 250 g/ℓ CrO_3 and 2.5 g/ℓ H_2SO_4 and a temperature of 55°C. The coatings were deposited on a polished flat sheet of annealed brass substrate and increased to a total chromium thickness of 75 μm which is optimum for x-ray diffraction analysis.

Determination of Optimum On/Off Duty Cycle

As discussed in the introduction, the effective interruption of current during chromium plating can produce crack-free deposits. This current interruption is responsible for disrupting the cathode film during chromium plating and thus can lead to changes in the deposit properties. The nature of this disruption during IC plating was investigated through polarization measurements using the same type of conventional chromic acid electrolyte at

55°C. The resulting current-voltage relationships shown in Figure 1 indicated that a current interruption of 15 sec duration was necessary to disrupt the cathode film completely. On the basis of these measurements, the on/off duty cycles of 30/5, 30/15, and 30/30 seconds were selected for studying the IC chromium deposits. Additionally, for each of the duty cycles indicated above, IC chromium deposits were prepared at current densities of 30, 45, and 60 A/dm^2.

STRUCTURAL RESULTS AND DISCUSSION

Appearance and Mechanical Properties

A comparison of some of the properties of IC and HC chromium is shown in Figure 2. The surface morphology of this IC chromium deposits was examined by means of a low magnification stereo microscope. The crack pattern of these deposits varied from a crack-free structure to one of heavy crack density. Corresponding to this structure, the hardness was found to vary between 770 and 1100 KHN and the tensile strength between 30.0 and 0 ksi. While a wide range of properties can be attained with IC chromium plating, better results are obtained from deposits plated at the higher current densities and longer interruption times.

Texture

The x-ray diffraction spectra shown in Figures 3 through 5 have been obtained by means of a diffractometer scan using molybdenum monochromatic radiation. For the sake of convenience these spectra have been organized in Figures 3 through 5 to reflect the changes which occur in the deposited structures as a result of the longer current interruption times and also the higher current densities. It is clearly seen that for the case of no current interruption, all three current density deposited structures are characterized by the expected and well known <111> fiber texture. When the current interruption time is increased, distinct differences in structure among the three current densities are found. For the case of the low current density of 30 A/dm^2 as seen in Figure 3, increasing the current interruption interval does not affect the characteristic (111) preferred orientation of the deposits. As shown in Figure 4 at 45 A/dm^2 a noticeable disturbance of the (111) preferred orientation occurred as the current was interrupted at the various duty cycles. As a result of the changes in intensities of the x-ray lines observed in Figure 4, it was deduced that a small fraction of the chromium crystallites grown in these deposits departed from the common <111> direction to random orientations and favored especially the <211> direction. As shown in Figure 5 at 60 A/dm^2, the current interruption produced deposits with a dual <211> and <111> fiber texture. Again from a comparison of the intensities observed the <211> texture of these deposits was stronger than the <111> texture, implying that a larger number of crystallites with the (211) rather than the (111) preferred orientation, have competitively grown parallel to the deposit surface. A comparison of intensities in Figures 4 and 5 showed that a greater fraction of the crystallites became random as the current density was increased from 45 to 60 A/dm^2.

Residual Stresses

Residual stresses in the IC chromium deposits were measured by means of an automated computer controlled x-ray diffraction system we developed and reported earlier.[7] Typically, the chromium deposit stresses varied from tensile stresses of 15 ksi to compressive stresses of 30 ksi. Regarding the stress measurements however, it should be noted that for the deposits with large microcrack densities the results were scattered and not always meaningful. The x-ray stress data had to be carefully interpreted to eliminate the effects of texture and high stress gradients that are present across the thickness of the deposits. In general, the high compressive stresses were measured from practically crack free samples that were deposited at the high interruption cycles and the current density of 60 A/dm^2.

Chromium Hydride Phase

As a consequence of the x-ray residual stress measurements it was found that an additional non-equatorial reflection occurred which did not belong in the bcc chromium structure, but satisfied the angular position of chromium hydride in the form CrH_x. Assuming that the chromium hydride phase has a preferred orientation as most other electrodeposits, this peak found off the equatorial direction provided a way of measuring qualitatively the retained chromium hydride in the IC chromium deposits. On the basis of the above findings our x-ray measurements on the IC chromium deposits show that CrH_x is almost always present at low current densities with no current interruption, and decreases significantly as the interruption time and current density reach their high values.

---

[7]G. P. Capsimalis, R. F. Haggerty, and K. Loomis, "Computer Controlled X-Ray Stress Analysis for Inspection of Manufactured Components," WVT-TR-77001, Watervliet Arsenal, Watervliet, NY, January 1977.

SUMMARY AND DISCUSSION

Several of our results are similar to those previously reported. In particular, the deposition of crack-free chromium by means of current interruptions is well documented.[1-4] The present study extends these earlier findings in providing more specific information on the effect of current interruption cycles and current density on the crystal structure and internal stress of the chromium deposits. While varying the on/off duty plating cycle and current density deposits with very different structures and properties have been observed. As a rule we found that increasing the current density to 60 A/dm^2 and the interruption interval to 30 sec have resulted in deposits with lower microcrack densities and improved mechanical properties. It was also found that uninterrupted deposits have a very strong <111> fiber texture while for high interruption times and 60 A/dm^2 current density, the <111> fiber texture is diminished considerably and the <211> orientation becomes dominant. Additionally, our measurements show that along with these changes there is a decrease in the chromium hydride retained in the deposit. The above evidence suggest a mechanism in which the crystallites with growth cones in the <111> direction preferentially trap the chromium hydride during coalescence. As the deposit becomes more random or tends towards the <211> texture less of the hydride phase is retained. The chromium hydride phase has a larger unit cell than bcc chromium and is unstable; and its decomposition

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[1]J. C. Saiddington and G. R. Hoey, Plating, 61, p. 923 (1974).
[2]H. Chessin and E. J. Seyb, Jr., Plating, 55, p. 821 (1968).
[3]H. Fry, Trans. Inst. Met. Finishing, 32, p. 107, (1955).
[4]W. Blum, Plating, 48, p. 613, (1961).

has been considered for a long time as being responsible for the presence of cracks in electrodeposited chromium.

Finally, the residual stresses measured were found to correlate best with electrodeposition rate due to current density and the orientation textures due to the interruption cycle.

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3. H. Fry, Trans. Inst. Met. Finishing, 32, p. 107 (1955).
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5. R. Weil, Plating, 57, p. 1231 (1970).
6. J. B. Kushner, Plating, 60, p. 1248 (1973).
7. G. P. Capsimalis, R. F. Haggerty, and K. Loomis, "Computer Controlled X-Ray Stress Analysis for Inspection of Manufactured Components," WVT-TR-77001, Watervliet Arsenal, Watervliet, NY, January 1977.

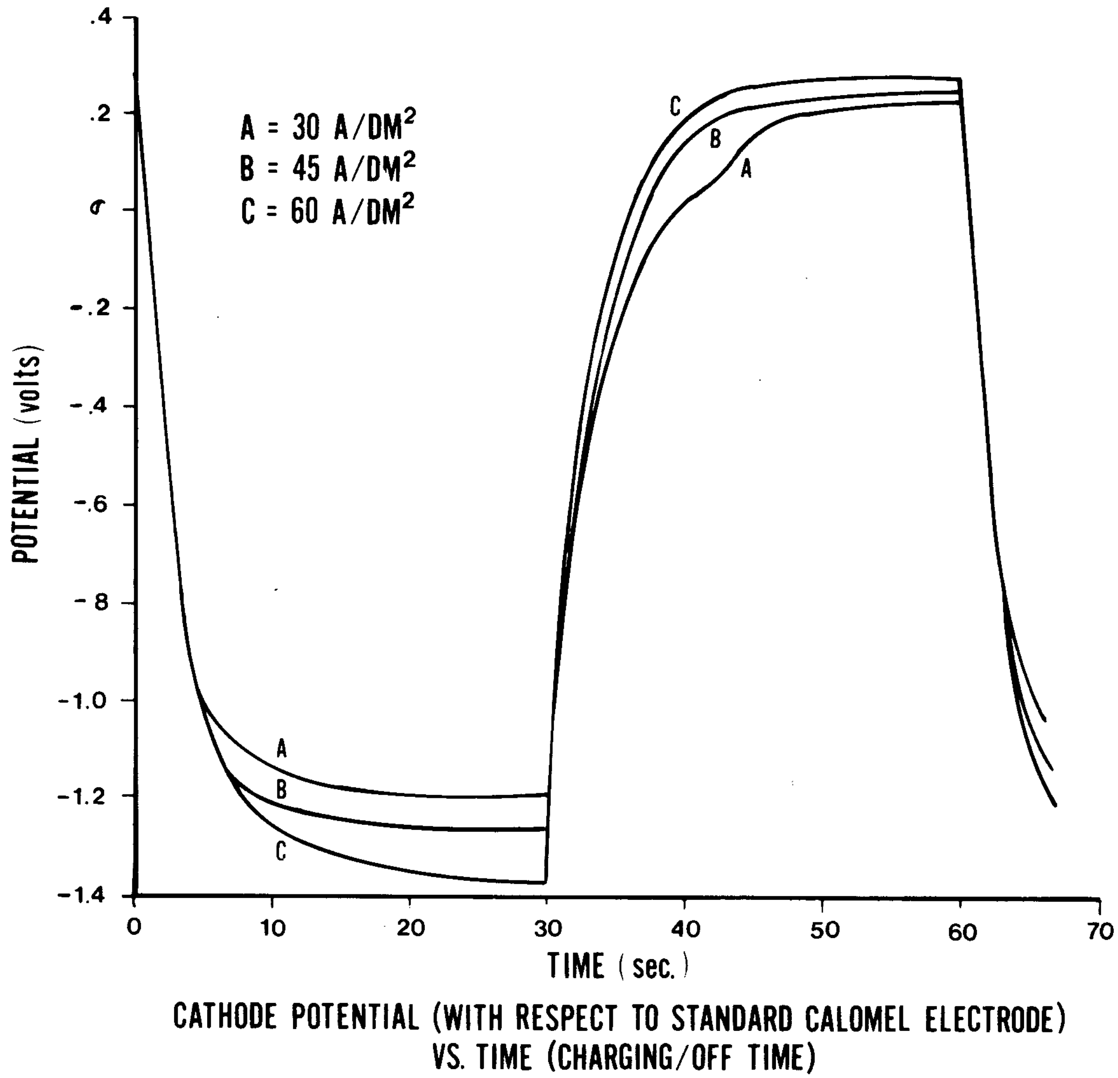
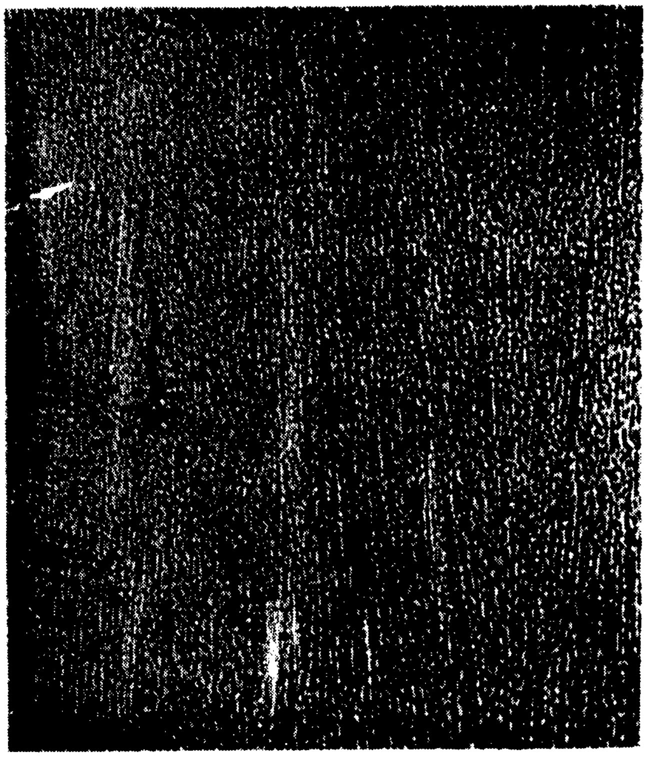
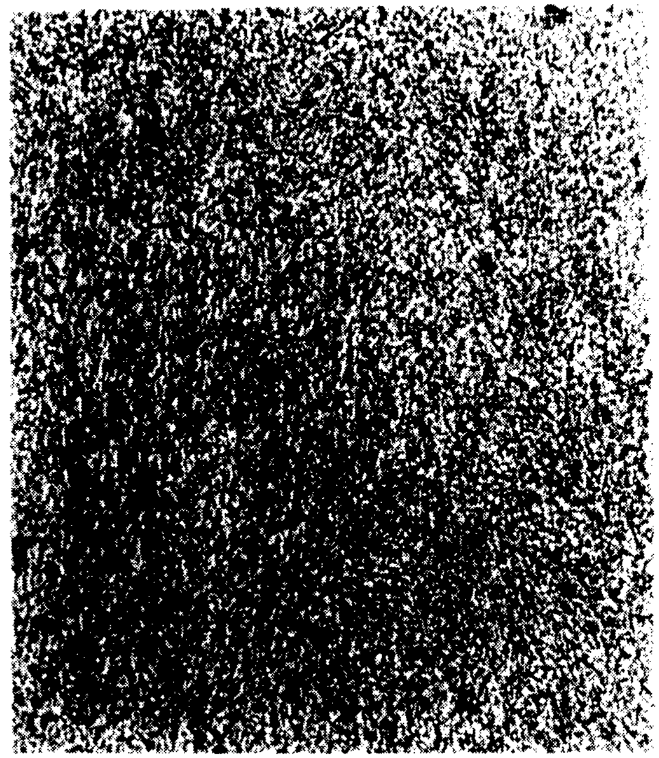


Figure 1. Cathode Potential(with respect to Standard Calomel Electrode) vs. Time (Charging /off Time).

STANDARD HARD CHROME (HC)	INTERRUPTED PLATED CHROME (IC)
	
1000X	1000X

	HC Chrome	IC Chrome
Bath Temperature °C	55	55
Current Density (A/dm^2)	30	35
Plating Rate (Mils/Hrs)	1	0.5
Hardness (KHN)	940	790
UTS (Psi)	15,000	30,000

Fig. 2. Comparison of Microstructure and Some Other Properties of HC and and IC Chromium.

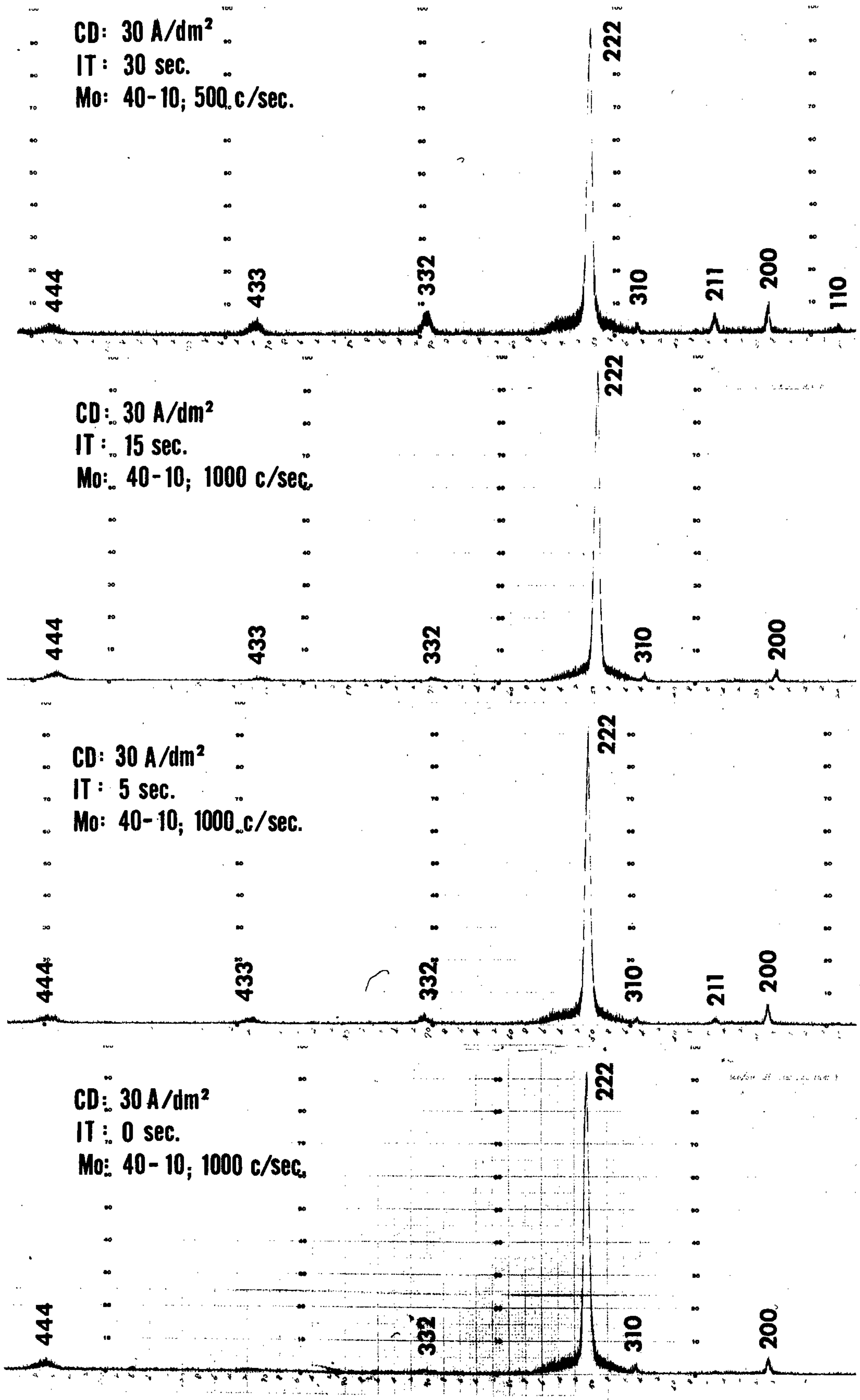


Fig. 3. X-Ray Diffraction Spectra of IC Chromium Plated at 30 A/dm^2 and Various Interrupt Time (IT) Intervals.

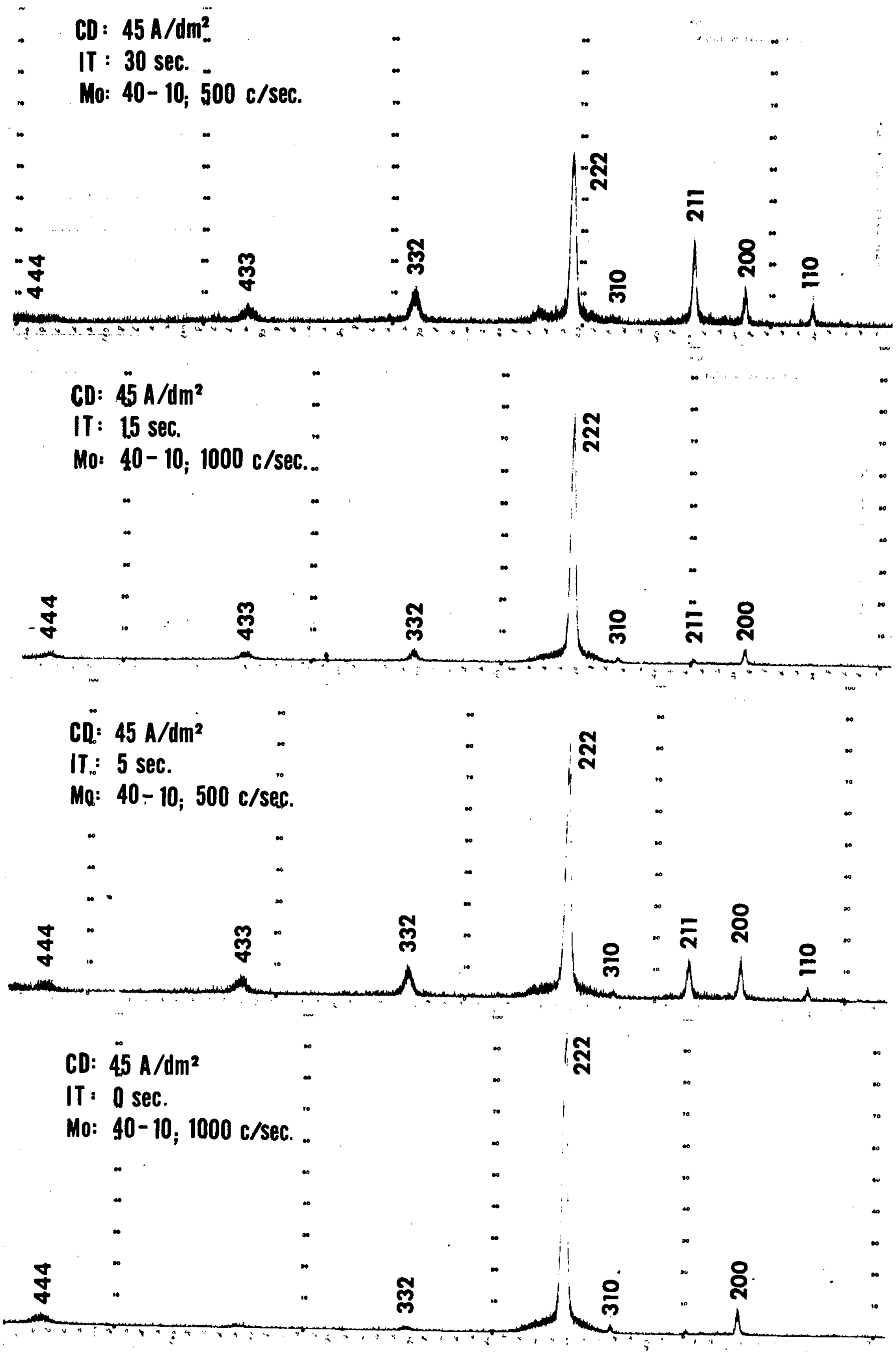


Fig. 4. X-Ray Diffraction Spectra of IC Chromium Plated at 45 A/dm^2 and Various Interrupt Time (IT) Intervals.

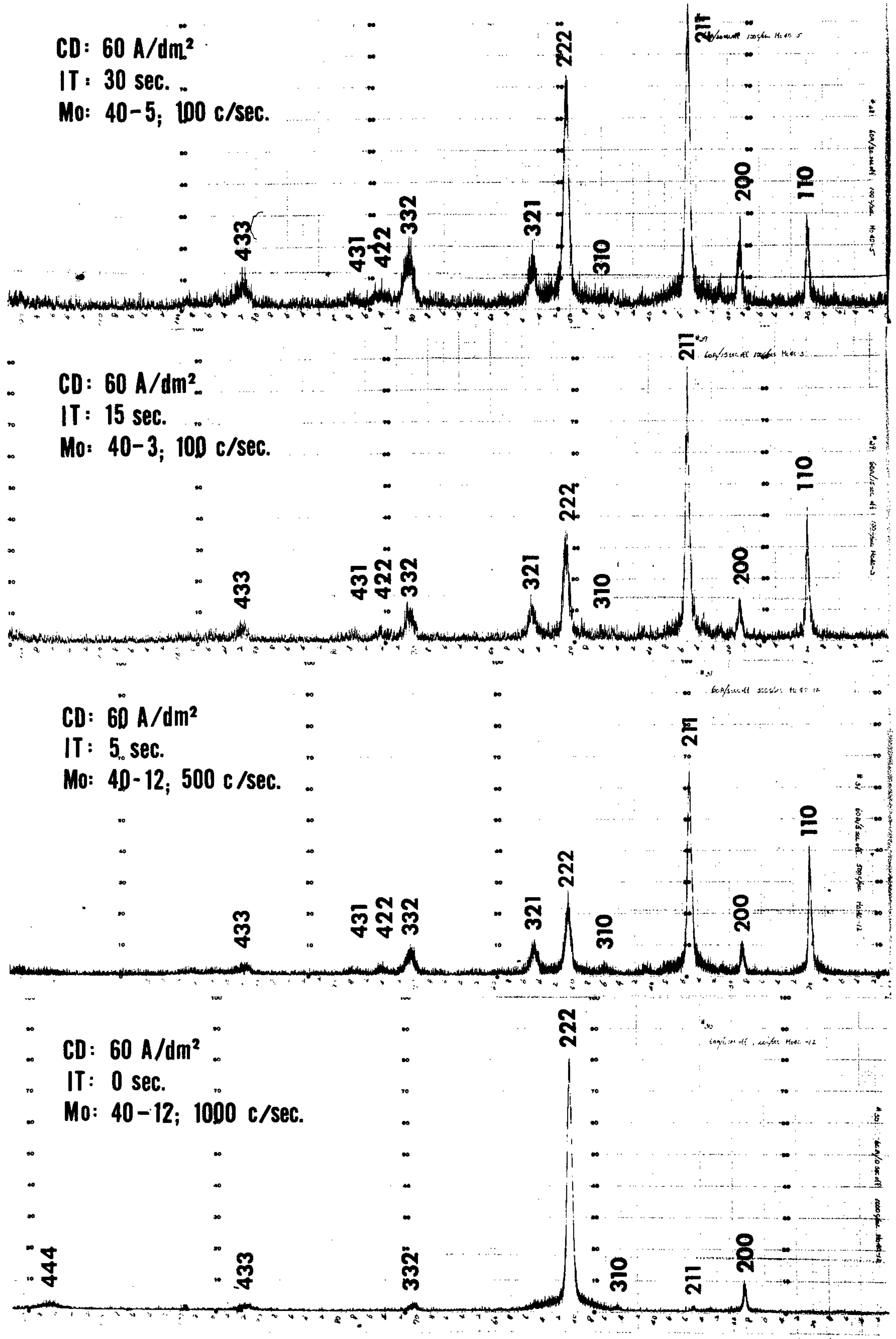


Fig. 5. X-Ray Diffraction Spectra of IC Chromium Plated at 60 A/dm^2 and at Various Interrupt Time (IT) Intervals.

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