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The Effects of Pulse Current Plating on the Mechanical Properties of Cobalt and Cobalt-Al₂O₃

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**The Effects of Pulse Current Plating on the
Mechanical Properties of Cobalt and Cobalt-Al₂O₃**

E. S. Chen and F. K. Sautter*

The Effects of Pulse Current Plating on the Mechanical Properties of Cobalt and Cobalt-Al₂O₃

E. S. Chen and F. K. Sautter*

ABSTRACT

The effect of high current pulses on the mechanical properties of electrodeposited cobalt and Co-Al₂O₃ was studied along with two other variables, ultrasonic agitation and superimposed direct current. The deposits were prepared at pulse current densities between 0 and 100 A/dm², a pulse width of 120 μsec and a pulse repetition time of 18 msec. The strength and hardness of both cobalt and Co-Al₂O₃ deposits made under these conditions showed substantial increases over conventionally prepared deposits. The improvement in mechanical properties is attributed to the refinement in grain structure and the enhancement of Al₂O₃ codeposition associated with the use of current pulses.

ELECTROPLATING WITH CURRENT PULSES is one of several variations of plating with modulated current which is finding increasing application for a variety of purposes. Burrus¹ employed high-voltage pulse plating to overcome problems associated with plating high-resistance or nonuniform-resistance surfaces of small area. Leidheiser and Ghuman² used pulse current plating to control deposit composition in the preparation of silver-tin alloys. Avila and Brown³ reviewed the advantages of pulse current plating which included dense deposits, increased plating rate and elimination of hydrogen embrittlement. Other advantages of plating with modulated current are cited in review articles published by Baeyens⁴ in 1954, by Dini⁵ in 1963 and by Cheh and Linford⁶ in 1974.

Our interest in the study of electroplating with high current pulses stemmed from our belief that this process may lead to electrodeposits with improved strengths. This belief is based on the premise that, because orientation in electrodeposits is dependent on the applied overvoltage⁷, the use of extremely high current pulses should change the deposit structure and consequently the deposit strength. The possibility of increasing the strength of electrodeposits by increasing the plating current density was suggested by Lamb⁸. However, he used only data from hardness measurements to conclude that the strength of copper prepared with high current pulses would be found to be in the ordinary range. The present work deals with the effects of high current pulses on the strength and microstructure of electrodeposited cobalt and dispersion-hardened cobalt-alumina alloys.

EXPERIMENTAL

Plating procedure

All chemicals were of reagent grade and used without further purification. A cobalt stock solution was prepared by dissolving 310 g/l CoSO₄ · 7H₂O, 41 g/l CoCl₂ · 6H₂O and 25 g/l H₃BO₃ in doubly distilled water. The stock solution was electrolyzed with corrugated cathodes at 0.2 to 0.4 A/dm² for 30 A-hr per litre and passed through 0.22 μ Millipore filters prior to use. Alon C (0.02 μ Al₂O₃, Cabot Corp) was added to the plating solution at 25 g/l in the preparation of dispersion-hardened Co-Al₂O₃ alloy samples.

The plating experiments were conducted in a 2-litre capacity Pyrex container using vertical brass cathodes, 40 cm² masked area, and rolled cobalt anodes hung in diaphragms made of filter paper. The plating solutions were stirred with a magnetically driven Teflon coated stirring bar. In addition, an ultrasonic generator (Sonogen Model AP-25B, Branson Ultrasonic Corp.) was used to minimize Al₂O₃ agglomeration in the plating suspension. The ultrasonic generator was operated at 40,000 Hz with a peak power output of 500 W.

Electrodeposits of cobalt and cobalt containing a dispersed Al₂O₃ phase were prepared at 40°C and pH 2.0 using d-c densities of 1.5-10 A/dm² with superimposed current pulses of 0-80 A/dm². A pulse width of 120 μsec and a pulse repetition of 18 msec were used. The pulse plating unit used is identical to that reported by Sullivan⁹. After electrodeposition, the brass substrate was dissolved anodically in a solution containing 100 g/l KOH and 50 g/l K₂CO₃. In this manner, electroformed cobalt and cobalt alloy sheets were obtained for testing.

Oxide determination

The amount of oxide in the deposits was determined gravimetrically. The samples were dissolved in 50 per cent HNO₃, the resulting suspension diluted with distilled water and the oxide separated with Millipore filters. The oxides together with the control filters were dried for 1 hr at 90°C and weighed on a microbalance.

Mechanical testing

Tensile test samples were prepared on a Tensile Kut Machine using a template with a 0.25-in. width and an 0.5-in. gauge length. An Instron Tensile Tester operated at a crosshead speed of 0.04 in./in./min was used to determine yield and ultimate tensile values.

Metallurgical examination

The cross section of plated samples was examined with a Leitz Metallograph Microscope. The microstructural features and oxide uniformity were brought out by etching in a 92 per cent HCl, 3 per cent HNO₃ and 5 per cent H₂SO₄ solution. Knoop Hardness values at a load capacity of 100 g were determined on this instrument fitted with a Leitz Hardness Tester.

RESULTS AND DISCUSSION

Pulse current plated cobalt

In the pulse current plating of cobalt, all of the deposits were prepared by using either: 1) high current pulses superimposed on a constant d-c background or 2) dc of different levels in conjunction with a constant pulse current density. Pulse current plating used in this manner is consistent with that defined by Baeyens⁴ and has the advantage of increased plating rate over ripple and interrupted current plating because of the absence of idle time.

Figure 1 shows the variations of strength and hardness in cobalt deposits as a function of current pulses superimposed on a background dc of 4 A/dm². These curves indicate that increasing the pulse current density produces a parallel increasing trend in hardness and strength reaching peak values at a pulse current density of 70 A/dm². At this point, the yield strength, hardness and tensile strength of the pulse plated

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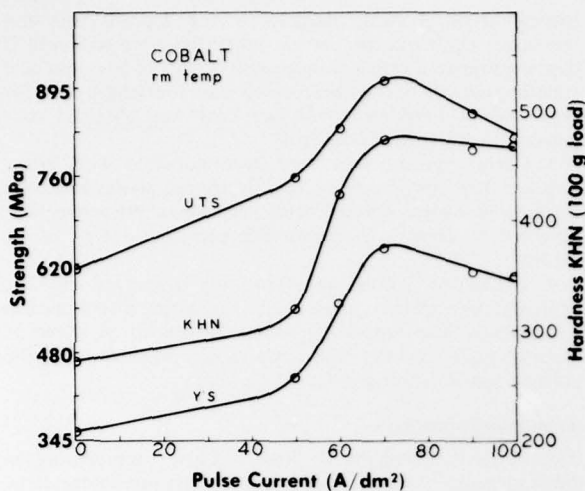
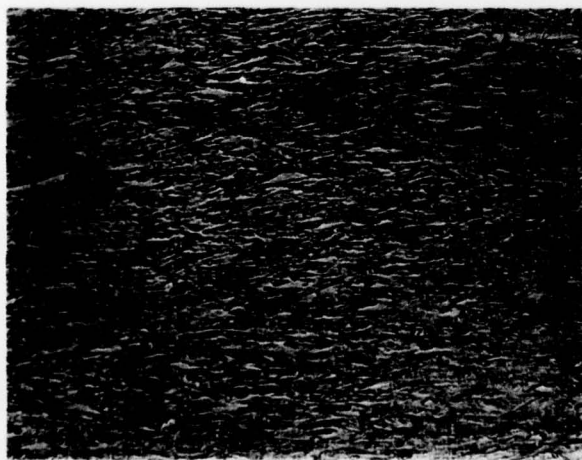


Fig. 1. Strength and hardness of cobalt electrodeposited as a function of pulse current density and dc at 4 A/dm^2 from a Watts electrolyte maintained at pH 2.0 and 40°C .



a.



b.

Fig. 2. Microstructure of cobalt electrodeposited from a Watts electrolyte maintained at pH 2.0 and 40°C . (a) continuous dc at 4 A/dm^2 , (b) pulse current at 70 A/dm^2 with superimposed dc at 4 A/dm^2 , original magnification 1,000x.

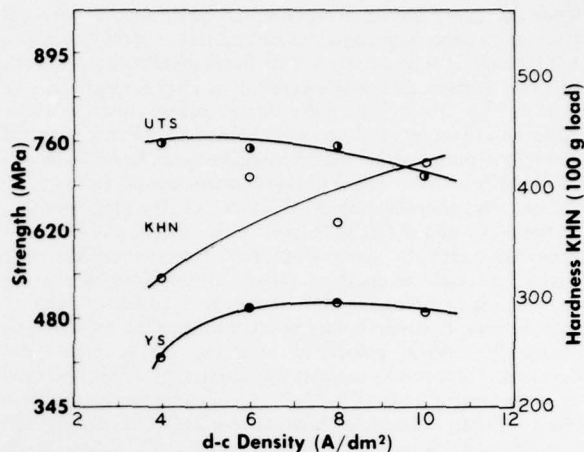


Fig. 3. Strength and hardness of cobalt electrodeposited as a function of d-c density at a pulse density of 50 A/dm^2 from a Watts electrolyte maintained at pH 2.0 and 40°C .

deposits show respective increases of 77, 67 and 48 per cent over conventionally prepared cobalt deposits. The subsequent decrease in strength for the deposits prepared above 70 A/dm^2 is suspect, and may be related to the observation that at 70 A/dm^2 , dendrites begin to form on the corners of the deposit, while above 70 A/dm^2 , dendritic formation occurs extensively on the corners and edges of the deposits. Consequently, the samples deposited above 70 A/dm^2 may have lower mechanical properties because the effective current densities are lower than the applied values.

The increases in hardness and strength of pulse current plated cobalt are attributed to the refinement in grain structure. Figures 2a and 2b show the microstructure of a cobalt deposit prepared with continuous dc at 4 A/dm^2 , and one with a pulse current density of 70 A/dm^2 superimposed on dc at 4 A/dm^2 . The refinement in grain size as illustrated in Fig. 2b is typical of the pulse current plated cobalt deposits.

Figure 3 shows the effects of increased dc used in conjunction with a constant pulse current density of 50 A/dm^2 . For the range of dc investigated, $4\text{--}10 \text{ A/dm}^2$, the curves indicate a slight increase in yield strength and hardness with increasing d-c

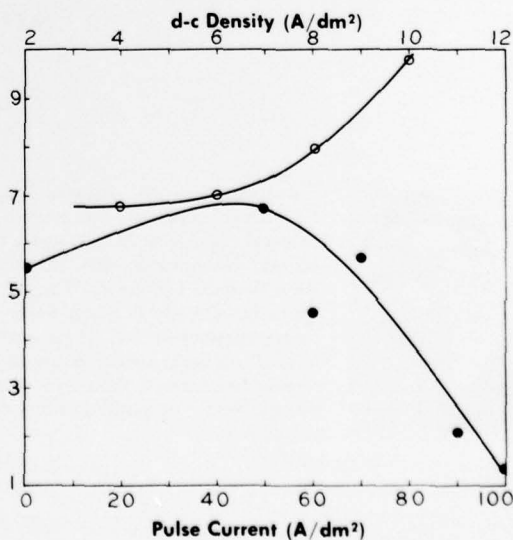
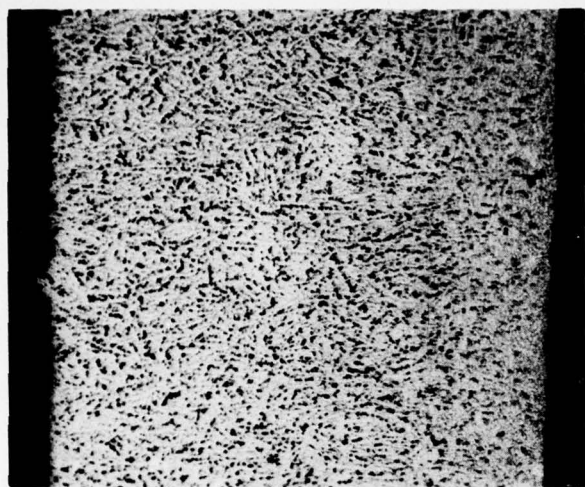


Fig. 4. Elongation in cobalt electrodeposited from a Watts electrolyte maintained at pH 2.0 and 40°C . Top curve (○)—as a function of d-c density and a pulse current density of 50 A/dm^2 . Bottom curve (●)—as a function of pulse current density and a d-c density of 4 A/dm^2 .



a.



b.

Fig. 5. Microstructure of Co-Al₂O₃ electrodeposited from a Watts electrolyte containing 25 g/l 0.02 μ Al₂O₃ using continuous dc at 4 A/dm², pH 2.0 and 40°C. (a) mechanical stirring, (b) ultrasonic agitation, original magnification 1,000x.

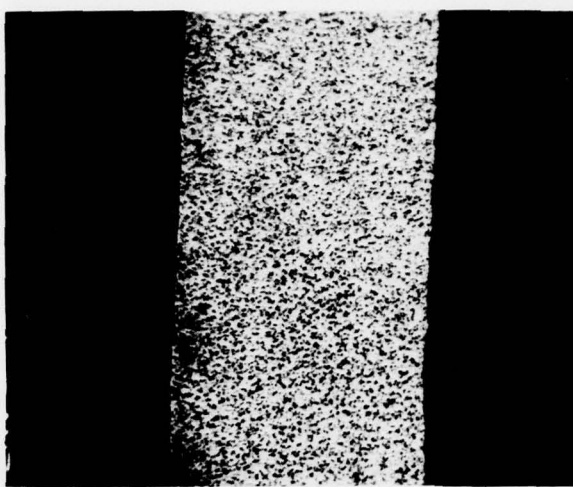
density. This trend was also observed by Sadak and Sautter¹⁰ in the conventional plating of cobalt from a Watts type electrolyte.

Figure 4 shows the elongation in the cobalt tensile samples. The top curve indicates that the elongation increases when the pulse current plated samples are prepared at higher background d-c densities. The lower curve shows an opposing trend in elongation for samples prepared with increasing pulse current density and constant background dc.

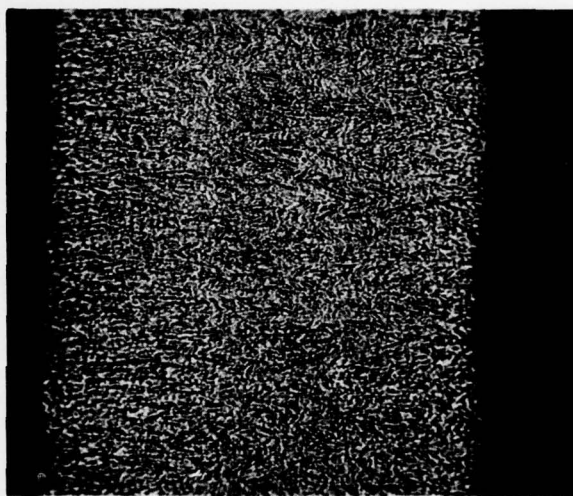
Pulse current plated Co-Al₂O₃

In the electrodeposition of dispersion-hardened Co-Al₂O₃, two plating parameters were selected for study. These were the effects of: 1) ultrasonic agitation and 2) pulse current plating on the mechanical properties of Co-Al₂O₃.

It is well known that the use of ultrasonic agitation in the plating of dispersion-hardened alloys will result in deposits with a more homogeneous distribution of the dispersed phase. This effect is illustrated in Figs. 5a and 5b both of which are photomicrographs of conventionally prepared Co-Al₂O₃; the former without and the latter with ultrasonic agitation. Similarly Figs. 6a and 6b show the microstructure of pulse current plated Co-Al₂O₃ without and with ultrasonic agitation respectively. In both instances, the use of ultrasonic agitation



a.



b.

Fig. 6. Microstructure of Co-Al₂O₃ electrodeposited from a Watts electrolyte containing 25 g/l 0.02 μ Al₂O₃ using pulse current at 50 A/dm² and superimposed dc at 4 A/dm², pH 2.0 and 40°C. (a) mechanical stirring, (b) ultrasonic agitation, original magnification 1,000x.

resulted in a more uniform distribution of the Al₂O₃ in the deposits. The increased uniformity of the oxide phase has essentially no influence on the mechanical properties of as plated Co-Al₂O₃. This is true for conventionally prepared Co-Al₂O₃¹⁰ as well as for pulse current plated Co-Al₂O₃ prepared without ultrasonic agitation, Figs. 7, 8 and 10 (dotted curves). These properties can be compared to the samples plated under the same conditions but using ultrasonic agitation (curves with open circles). For all practical purposes, the mechanical properties in the deposits prepared with and without ultrasonic agitation are quite similar. Nevertheless, it is expected that Co-Al₂O₃ deposits with a more homogeneous dispersed phase will show improved high temperature properties, and this will be described in a later report.

Figure 10 shows the codeposition of Al₂O₃ in cobalt as a function of pulse current density and background d-c density. These values may be compared with the 1.3 vol per cent Al₂O₃ obtained in the conventional plating of Co-Al₂O₃ using continuous dc at 4 A/dm². It is interesting to note that below a background d-c level of 6 A/dm², pulse current plating introduces more Al₂O₃ into the deposits as compared to those produced conventionally, while above 6 A/dm², the incorporation of Al₂O₃ is less.

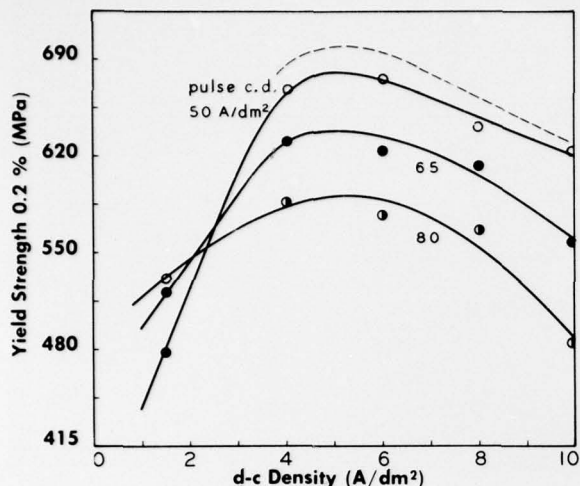


Fig. 7. Yield strength of Co-Al₂O₃ electrodeposited from a Watts electrolyte containing 0.02 μ Al₂O₃ maintained at pH 2.0 and 40°C. Dashed curve—plated with pulse current at 50 A/dm², no ultrasonic agitation. (○)—plated with pulse current at 50 A/dm² with ultrasonic agitation. (●)—plated with pulse current at 65 A/dm² with ultrasonic agitation. (⊙)—plated with pulse current at 80 A/dm² with ultrasonic agitation.

It was mentioned earlier that dendritic formation in pulse current plated cobalt may be responsible for the lowering of strength in the cobalt deposits prepared at pulse current densities above 70 A/dm². A similar reasoning may be applied to Co-Al₂O₃ to account for the decrease in strength associated with the deposits prepared at pulse current densities of 50, 65 and 80 A/dm². For all levels of background d-c densities investigated, the Co-Al₂O₃ deposits prepared with superimposed current pulses of 50 A/dm² showed no dendritic formation, while at 65 A/dm², dendritic formation occurred on the corners of the deposits, and at 80 A/dm², dendritic growth formed extensively on the corners and edges of the deposits. Metallographic attempts to bring out the grain structure in these deposits were unsuccessful because the grain boundaries are masked by preferential etching around the Al₂O₃ particles (see Figs. 6a and 6b).

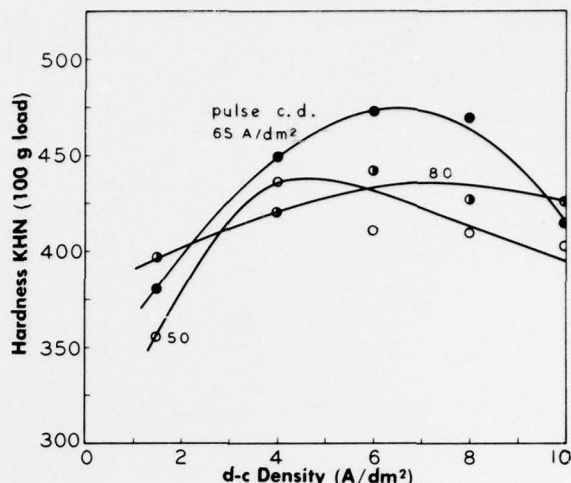


Fig. 9. Knoop hardness of Co-Al₂O₃ electrodeposited with ultrasonic agitation from a Watts electrolyte containing 0.02 μ Al₂O₃ maintained at pH 2.0 and 40°C. (○)—plated with pulse current at 50 A/dm². (●)—plated with pulse current at 65 A/dm². (⊙)—plated with pulse current at 80 A/dm².

The yield strength, tensile strength and hardness of pulse current plated Co-Al₂O₃ deposits are shown in Figs. 7, 8 and 9. In comparing these values, reference must be made to the oxide content as shown in Fig. 10 for the plating conditions used. In view of the relatively large spread in codeposited Al₂O₃, 2.0 to 0.5 vol per cent, it would be desirable to compare the mechanical properties of these deposits by normalizing the Al₂O₃ content to a specific value. However, normalization of the present data is impractical because it is now necessary to consider how each of two parameters, pulse current and oxide content, can interact to affect the mechanical properties. The effects of these two parameters on yield strength can be seen by comparing Figs. 1 and 7 for cobalt and Co-Al₂O₃. For the deposits prepared at current pulses of 50, 65 and 80 A/dm² with dc at 4 A/dm², the yield strengths are 420, 605 and 620 MPa for cobalt and 670, 625 and 585 MPa for Co-Al₂O₃ respectively.

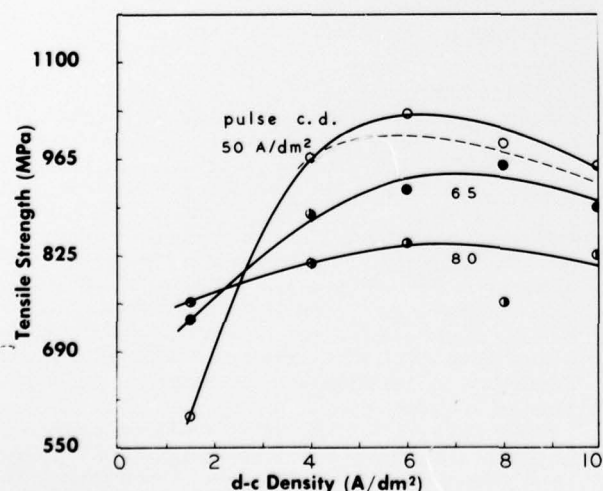


Fig. 8. Tensile strength of Co-Al₂O₃ electrodeposited from a Watts electrolyte containing 0.02 μ Al₂O₃ maintained at pH 2.0 and 40°C. Dashed curve—plated with pulse current at 50 A/dm², no ultrasonic agitation. (○)—plated with pulse current at 50 A/dm² with ultrasonic agitation. (●)—plated with pulse current at 65 A/dm² with ultrasonic agitation. (⊙)—plated with pulse current at 80 A/dm² with ultrasonic agitation.

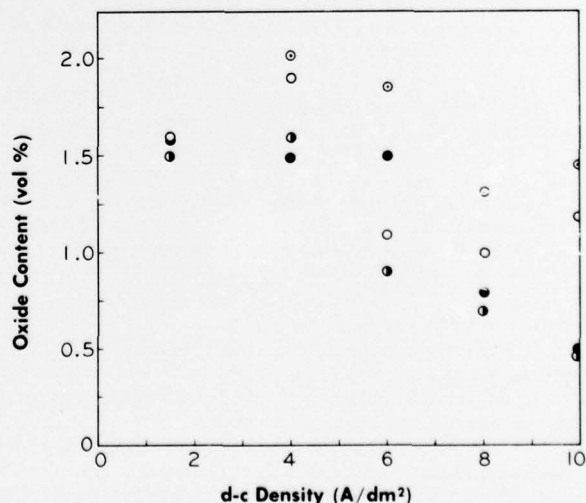


Fig. 10. Vol per cent Al₂O₃ codeposited from a Watts electrolyte containing 0.02 μ Al₂O₃ maintained at pH 2.0 and 40°C. (○)—plated with pulse current at 50 A/dm², no ultrasonic agitation. (○)—plated with pulse current at 50 A/dm² with ultrasonic agitation. (●)—plated with pulse current at 65 A/dm² with ultrasonic agitation. (⊙)—plated with pulse current at 80 A/dm² with ultrasonic agitation.

Thus the incorporation of Al_2O_3 into cobalt improves the yield strength in deposits prepared at current pulses of 50 A/dm^2 but has little effect at 65 and 80 A/dm^2 . Similarly, the contributions of pulse current and Al_2O_3 content to yield strength can be seen in Figs. 3 and 7 for pulse current plated cobalt and Co- Al_2O_3 as a function of d-c density. According to Fig. 3, the yield strength in pulse current plated cobalt becomes higher from 420 to 495 MPa with increasing background d-c density from 4 to 10 A/dm^2 . By incorporating Al_2O_3 in cobalt, the yield strength increases by about 170 MPa over pulse current plated cobalt prepared under the same conditions. However, unlike pure cobalt, the yield strength in pulse current plated Co- Al_2O_3 decreases with increasing current density from 4 to 10 A/dm^2 . The decrease in strength may be attributed to the reduction in codeposited Al_2O_3 associated with the use of higher d-c densities. These results indicate that the strengthening observed in pulse current plated Co- Al_2O_3 is related to two cooperative effects. Firstly, the use of high current pulses in plating will intensify nucleation and growth processes leading to a refinement in grain structure. Secondly, increasing the dispersed phase has the effect of increasing the deposit defect structure. Both grain refinement and increased dispersed phase can increase the strength by imposing more resistance to oppose dislocation motion during deformation¹¹. It is difficult to ascertain which of the two effects plays the dominant role in improving the strength of pulse current plated Co- Al_2O_3 deposits. However, the indications are that the incorporation of Al_2O_3 in pulse current plated cobalt will improve the strength only when the strength in these deposits has not already been optimized by the use of high current pulses.

SUMMARY

1. The use of high current pulses can increase the strength, improve the hardness and refine the microstructure in electrodeposited cobalt. The highest strength increase, 77 per cent over conventionally prepared cobalt was obtained with the use of a pulse current density of 70 A/dm^2 and a superimposed dc of 4 A/dm^2 . Dendritic formation appeared with the use of current pulses above 70 A/dm^2 .

2. The mechanical properties of Co- Al_2O_3 , as in cobalt, were also improved with the use of pulse current plating. The

incorporation of oxide particles in the deposits was also increased. Optimum mechanical properties were produced in deposits prepared with a pulse current density of 50 A/dm^2 and superimposed dc of 6 A/dm^2 . An increase in strength of 91 per cent and in hardness of 54 per cent was obtained over conventional Co- Al_2O_3 deposits.

3. Current pulses were used in conjunction with superimposed direct current to increase the overall plating rate. At a given pulse current density, better mechanical properties were obtained in deposits prepared with superimposed dc of $4-6 \text{ A/dm}^2$.

4. Ultrasonic agitation effectively dispersed particle agglomeration and produced a uniform particle distribution in the deposits. The importance of this feature will be shown in ensuing studies on the high temperature properties of pulse current plated cobalt and Co- Al_2O_3 alloys.

ACKNOWLEDGMENTS

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Discussions of this paper are invited for publication in a future issue of *This Journal*.

MEET THE AUTHORS



EDWARD S. CHEN attended Rensselaer Polytechnic Institute where he received a BS in chemical engineering in 1959 and a PhD in physical chemistry in 1964. He joined the Benet Weapons Laboratory, Watervliet, N.Y. where he is presently group leader of the electrodeposition section. He is involved in research on the electrodeposition of dispersion-hardened alloys, the effects of process variables on the properties of electrodeposits and adhesion.

FRITZ K. SAUTTER received a PhD in metallurgy from the University of Stuttgart in 1955. He has worked with Professor Raub in Schwabisch Gmund as research assistant on the electrodeposition of alloys. Prior to joining Watervliet Arsenal in 1959, he was employed with DEGUSSA, Hanau, and with Riedel and Co. in Bielefeld. In 1963 he received the U.S. Army R and D Achievement Award for his contributions to the electrodeposition of dispersion hardened alloys. Since 1971 he is Chief of the Physical Science Division at Watervliet Arsenal.



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