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The Generation of Cobalt Emitter for Field Ionization

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ABSTRACT. A new method for the preparation of cobalt dendrites on tungsten wires is described. A simple drop cell is constructed. DC pulses through the cell proved to be unsuitable for the production of cobalt dendritic surfaces. Crystalline cobalt dendrites are obtained by using AC pulses. The cobalt emitter is examined as a field anode in the field ionization source.

Introduction

Single tips are used as emitters in field of microscopy[1,2]. Conditioned tungsten wires find much wider use because they offer larger emission currents[3,4]. Bursey *et al.*[5-7] prepared field emitters by cathodic metal deposition on tungsten wires from aqueous solutions of simple salts by using a pulse generator connected with a DC power supply.

Goldenfeld and Veith[8] used a drop cell method in conjunction with a DC supply to prepare dendritic emitters. The main disadvantage of their method is the probability of destruction of the dendrites while removing the emitter from the cell. Recently, Zahran *et al.*[9] used, for the first time, normal sine waves in the drop cell method.

The aim of the present paper is to carry out a study using the newly developed drop cell technique[9] to prepare a dendritic emitter surface of cobalt.

Experimental

Ten μm tungsten wire is spot welded onto the emitter supports. The supports are fixed in a movable vertical column. The electrodeposition of cobalt dendrites is per-

formed in the following way. A drop of 0.5 m $Co(NO_3)_2$ solution is placed between two fixed platinum ribbons 0.5 mm in thickness and 1 mm apart by using a syringe. Then, by the aid of a microscope, the 10 μm wire is adjusted inside the drop to the required position. The electric circuit used for the electrodeposition of cobalt dendrites is shown in Fig. 1. A time gate is connected to the sine wave supply, and a selected number of sine waves can be controlled. The current strength through the electrolytic cell is measured by an oscilloscope connected across a sampling resistor.

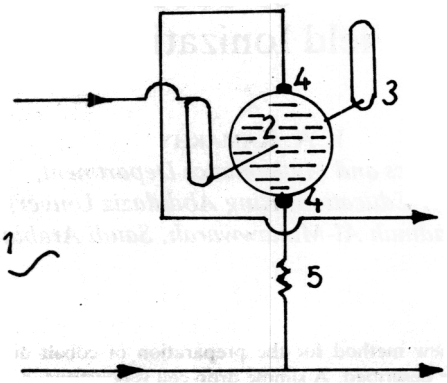

FIG Electric circuit used for the electrodeposition of cobalt on tungsten.
1. 50 hz sine wave.
2. Drop of $Co(NO_3)_2$ solution.
3. 10 μm tungsten wire.
4. Platinum electrodes.
5. Sampling resistor connected to an oscilloscope.

Results and Discussion

Trials were attempted for electrodeposition of metallic cobalt from $Co(NO_3)_2$ aqueous solution drop on tungsten wire using either DC or AC power sources. The deposits obtained were examined by a scanning electron microscope and the corresponding electron micrographs (Figs. 2 and 3 respectively) showed two morphological structures. As can be seen in Fig. 2, the use of 7 DC pulses at 2.5 V and pulse duration 5×10^{-2} sec. leads to the production of coarse-grained cobalt deposit. On the other hand, by using an AC source of the normal 50 Hz sine wave at 2.5 V for 3 minutes a finer-grained cobalt deposit is produced as shown in Fig. 3.

The AC pulse shape, as observed through a sampling resistor connected to an oscilloscope during electrodeposition, is changed from a sine wave to a partially rectified one (Fig. 4). The theoretical foundation of AC electrolysis was first considered by Warburg[10]. Doss and Agrawal[11,12] reported that when an alternating field

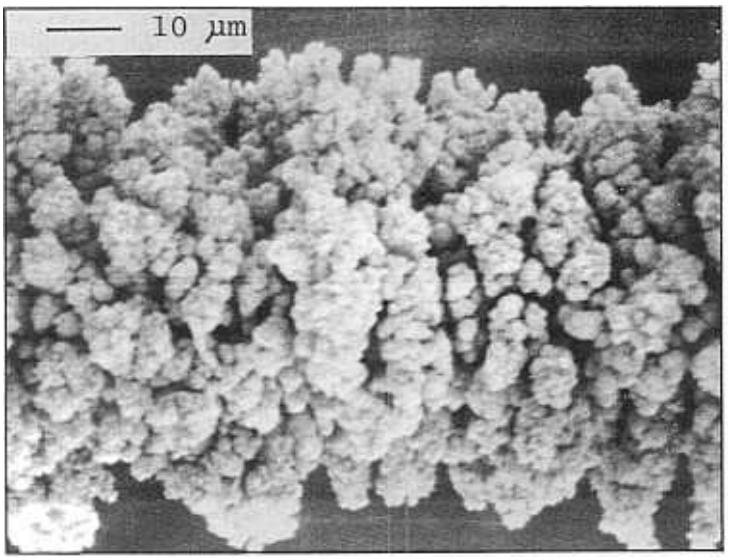

FIG. 2. Electron micrograph of Co layer on tungsten wire after 5 pulses at 2.5 V DC (× 1000).

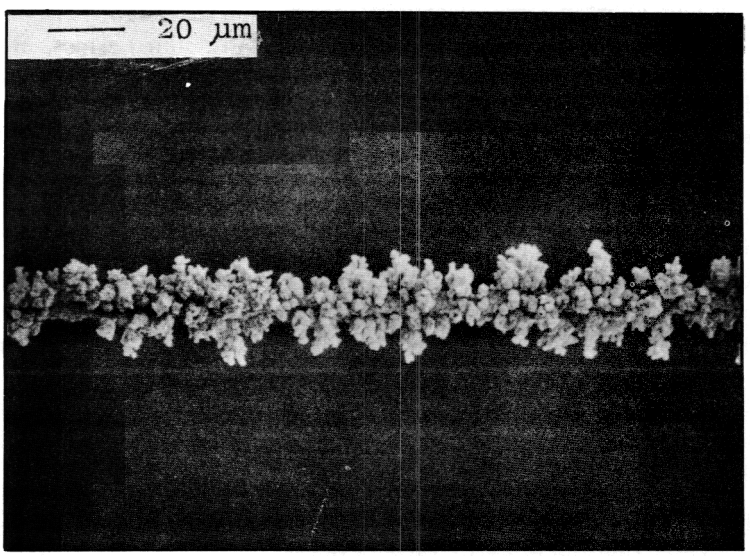

FIG. 3. Electron micrograph of Co dendrites on tungsten after 3 min. at 2.5 V AC (× 500).

was connected to two platinum electrodes dipped in an aqueous electrolytic system, a DC potential could be detected at each electrode. Later, this experimental fact was well defined as a faradaic rectification phenomenon by Oldham[13] and by Fleischman and Oldham[14].

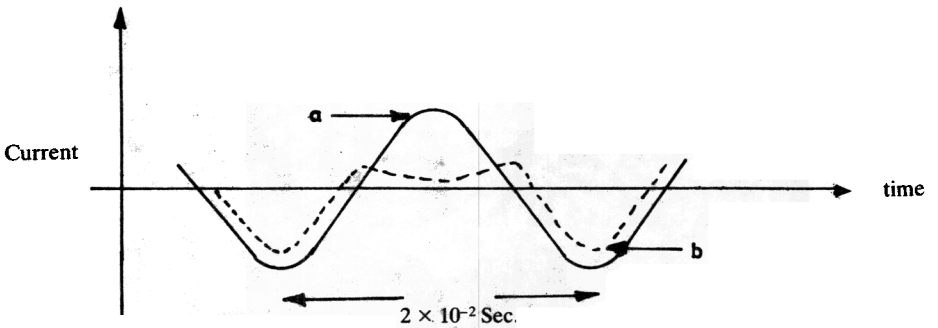

FIG. 4. Changes in the AC pulse shape during electrodeposition.
a. The applied pulse.
b. The rectified pulse.

It is observed also, that the time of deposition of the coarser grained deposit (3.5×10^{-1} sec.) is very short in comparison with that needed for the formation of the finer grained deposit during the cathodic half wave of AC (1.5 min.). This is in agreement with Wranglen's observation[15] that redissolution of the cathode deposits often occurred during deposition with periodically reversed current in the solution. The redissolution phenomenon means a reversed current *i.e.*, the net result is a low current density.

Similarly, in the drop cell electrolyte, dissolution can occur during the anodic half wave. Therefore, different crystalline pits could be formed and irregular rolling-off layers occurred, forming a rough surface. During the following cathodic half wave the surface is partially healed so that its roughness becomes permanent. During the next anodic half wave some crystals are partially dissolved and at the beginning of the cathodic half wave numerous new crystals could be formed. The rate of crystal growth during the cathodic half waves is faster than that of dissolution during the anodic half waves, as can be concluded from the pulse shape. This is in accord with the faradaic rectification phenomenon.

Field ionization mass spectrum of n-heptane taken with cobalt emitter is shown in Fig. 5. It is know that the intensity ratio of the fragment ion $C_2H_5^+$ to the molecular ion $C_7H_{16}^+$ in the field ionization spectrum of n-heptane characterises the field strength[16]. With the present emitter the intensity ratio is about 2×10^{-2} which means, according to Speier *et al.*[16], a field strength of about $1\text{-}2 \times 10^7$ V/cm.

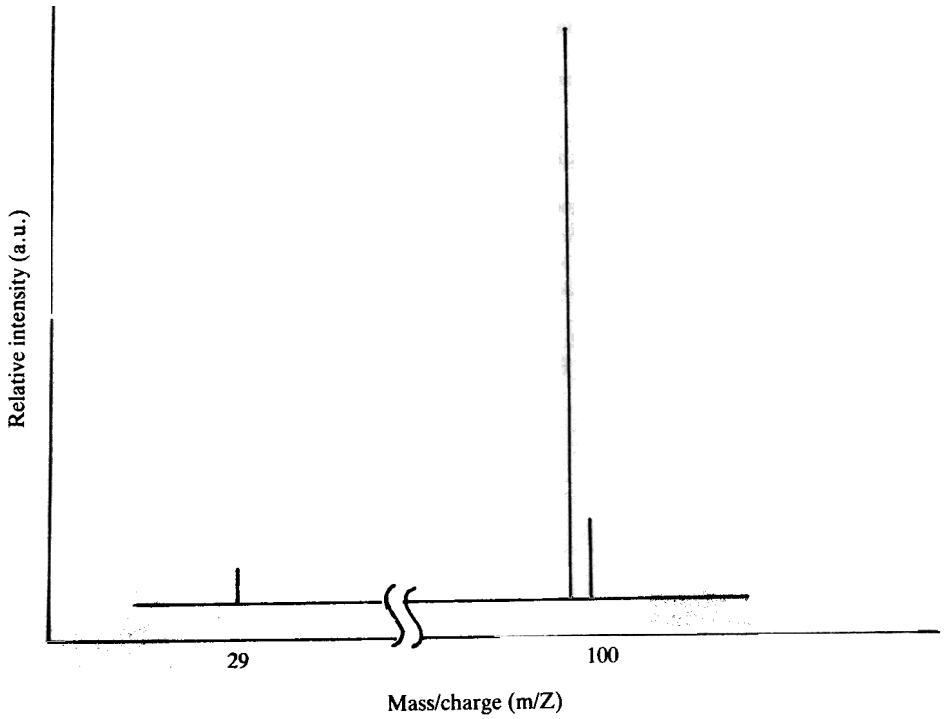

FIG. 5. The field ionization mass spectrum of n-heptane using cobalt emitter.

After production of the cobalt emitter, a very weak ion emission is observed at room temperature. A high emission is achieved only after thermal treatment of the emitter up to about 600°C.

Acknowledgement

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إنتـــاج مصاعـــد الانبعاث الأيـــوني المجالي من الكـــوبالت

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المستخلص . هناك طريقة حديثة لتحضير مصاعد لها زوائد متشجرة من الكوبالت على سطح سلك من التنجستين تختلف عن الطريقة التقليدية . وقد تم بناء الخلية بطريقة سهلة وذلك بتثبيت نقطة من محلول نترات الكوبالت بين سلكين رفيعين من البلاتين .

ولقد ثبت أن الطريقة التقليدية والتي يوضع فيها الجهد الثابت بين القطبين لا تصلح لإنتاج الزوائد المتشجرة في الخلية بينما اتضح أن تكوين بلورات الكوبالت يمكن حدوثه عند استخدام نبضات من مصدر تيار متردد يمكن التحكم في عدد نبضاته .

ولقد تم اختيار المصعد الناتج من بلورات الكوبالت على التنجستين في المنبع الأيوني المجالي وكذلك تمت مناقشة الظروف المنافسة لإنتاج الطيف لجزئيات الهبتان العادي في مطياف الكتلة باستخدام المصعد الأيونى الجديد .