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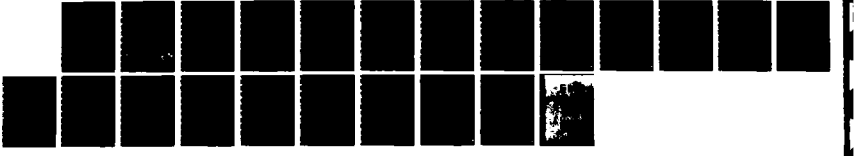

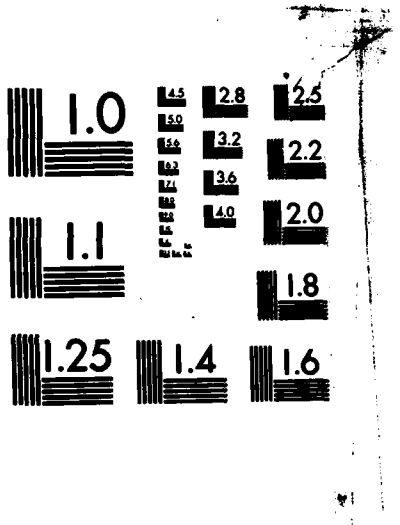

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REPORT SD-TR-83-96

Auger Characterization of New and Aged Dispenser Cathode Surfaces

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This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

Mark L Warenda

Mark L. Warenda, 1st Lt, USAF
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The major changes in dispenser cathode surface signatures caused by aging were studied on B-type dispenser cathodes by operating them at high temperatures. By means of a computerized scanning Auger microprobe (SAM), the surface characteristics of three aged cathodes were measured and compared with those of a new cathode. Several hundred points were monitored on each sample to develop a good statistical description of each cathode surface, including both the impregnated pore ends and the tungsten regions

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between the pores. A method of discrimination between impregnant regions and tungsten regions has been developed, based on differences in the oxygen Auger peak shape and position. The results show that a surprisingly large fraction (>70%) of the new cathode surface could be similar to the impregnant. This fraction decreases to near 20% with age. The elemental composition of these different regions was examined by correlating the presence of each element to others. The elemental correlation showed that the dominant fraction of the cathode surface in early life is similar to a barium-oxide on tungsten, and the cathode changes to an increasing fraction of surface tungsten oxide with aging.

PREFACE

We wish to thank R. L. Corbin and R. Harrell for designing and building the hardware interface linking the SAM to the computer, H. R. Hedgpeth for designing and implementing the software link between the data analysis routines and the SAM, and B. Lin for assistance with the graphics development.

CONTENTS

FIGURES

I. INTRODUCTION

Dispenser cathodes are high current density electron emission devices that are fabricated by impregnating a porous tungsten matrix with a mixture of barium calcium aluminates.[1] During operation, barium continually migrates from the impregnated pores onto the cathode upper surface, lowering the work function and giving rise to copious electron emission.[2-4] Since cathode surfaces consist of an active monolayer of barium and oxygen on tungsten[5-7] for electron emission, electron microprobe instruments are ideally suited for dispenser cathode analyses. Much work has been done on dispenser cathodes using surface analysis techniques, but, to our knowledge, very few surface statistical studies of new and aged dispenser cathodes have been made. This report presents results obtained by using a modified scanning Auger microprobe (SAM) to compare the emissive surface of new and thermally aged dispenser cathodes.

II. EXPERIMENTAL CONFIGURATION

B-type dispenser cathodes, made from 82 to 84% dense tungsten billets and using a 5:3:2 impregnant (mole ratios of BaO, CaO, and Al_2O_3) were obtained from Spectra-Mat, Inc., Watsonville, California. Three samples were activated by raising and holding the temperature for 1 hr at 1191°C. The temperature was measured using a disappearing filament optical pyrometer. The samples were then aged at 1086, 1191, and 1296°C, respectively, for 1500 hr in a vacuum of 2×10^{-9} Torr. All temperatures were corrected both for window absorptance and for deviation from ideal black body behavior by means of a correction determined by Longo:[8]

Temp (°C, true) = [Temp (°C, brightness)-14.2]/0.9536

The aged cathodes and a new cathode were then transferred into the SAM analysis chamber,[9] and reactivated in situ prior to analysis. The 1296°C-aged sample could not be reactivated because of heater filament degradation during aging. After reactivation, the new cathode and the 1086 and 1191°C aged cathodes were each analyzed at 1000 ± 3°C, 1102 ± 2°C, and 1205 ± 5°C, after being held at least 24 hr at each temperature for surface equilibration. The three different ranges of analysis temperatures indicate the changes that may occur if the aged cathodes operate at different temperatures. Aging the cathodes for 1500 hr at different temperatures yields differing amounts of accelerated aging. The data taken using the 1102°C analysis temperatures most closely represent cathode aging under normal operating conditions (1102°C true = 1053°C uncorrected).

The SAM was interfaced with an HP-1000 minicomputer for data acquisition and analysis. At each spatial point, this system automatically determined the Auger peak-to-peak height and the energies associated with each Auger peak. It could also store the signal-versus-energy spectrum for any given element. Several hundred spatial points within a 100 × 100 μm viewing area were analyzed on each sample to obtain a statistically representative description of the cathode surface. The major Auger signals monitored were the barium (600 eV), oxygen (~503 eV), and tungsten (179 eV) peaks. The low-energy

179 eV tungsten peak was used instead of the tungsten 1736 eV peak, to maximize the surface sensitivity. It also allowed the use of a low (3 kV) beam voltage to minimize electron beam induced desorption. Surface impurities, such as carbon, evaporated at temperatures below 935°C, which was the lowest analysis temperature used. Residual sulfur was also seen as a surface impurity, but its presence could not be correlated well to either barium, oxygen, or tungsten. The cathodes were examined using a 2-μm diameter beam, and a time constant of 0.3 to 1.0 sec. Elemental concentrations were determined from signal peak-to-peak amplitudes, and were adjusted for Auger cross section and beam voltage using the handbook[10] elemental sensitivities. This gives semi-quantitative results, but it does not correct for possible sample-specific matrix effects.[11]

III. RESULTS

The dispenser cathode is a spatially inhomogeneous device, consisting of exposed pore ends and tungsten grain regions. The tungsten regions between the pores are covered with a monolayer or submonolayer of barium and oxygen.[5-7] We have developed an effective means of discrimination between these two types of regions using the shape of the oxygen Auger peak. Figure 1 shows a plot of the width of the oxygen Auger peak (difference in energy between the down-swing and up-swing in dN/dE-mode) versus energy (down-swing) of the peak. These data were taken on a preselected pore and tungsten region point as the cathode temperature was raised during reactivation. Part of the observed differences between the pore and the tungsten regions in Fig. 1 may be due to "charging," and part may be due to the different oxygen chemical environment in the two regions. However, the oxygen signal characteristics, as shown in Fig. 1, were found to be reproducible enough to be used as a sensitive surface discrimination tool, separating the surface into impregnant-like and tungsten-like regions.

Using a plot similar to Fig. 1, the data from the new and aged cathode were sorted by ascribing the oxygen signal with both a peak width <9.0 eV and peak energies >512 eV to tungsten regions, and ascribing the remainder to pore or impregnant-like regions. This gave fractional surface coverages as shown in Table 1, while operating the new and aged cathodes at 1102 ± 2°C. These data show that, in early life, cathode surfaces are dominated by the impregnant-like regions, and this fraction decreases with aging. But even in the 1086°C aged sample, the impregnant-like fraction remains higher than the exposed pore-end fraction (16 to 18% for an 82 to 84% dense tungsten billet material), which demonstrates that these regions remain beyond a normal 1000-hr cathode burn-in period.

Table 1. Cathode Surface Coverage

	Impregnant regions	Tungsten regions
New cathode	77.6%	22.4%
Cathode aged 1500 hr at 1086°C	56.6%	43.4%
Cathode aged 1500 hr at 1191°C	24.2%	75.8%

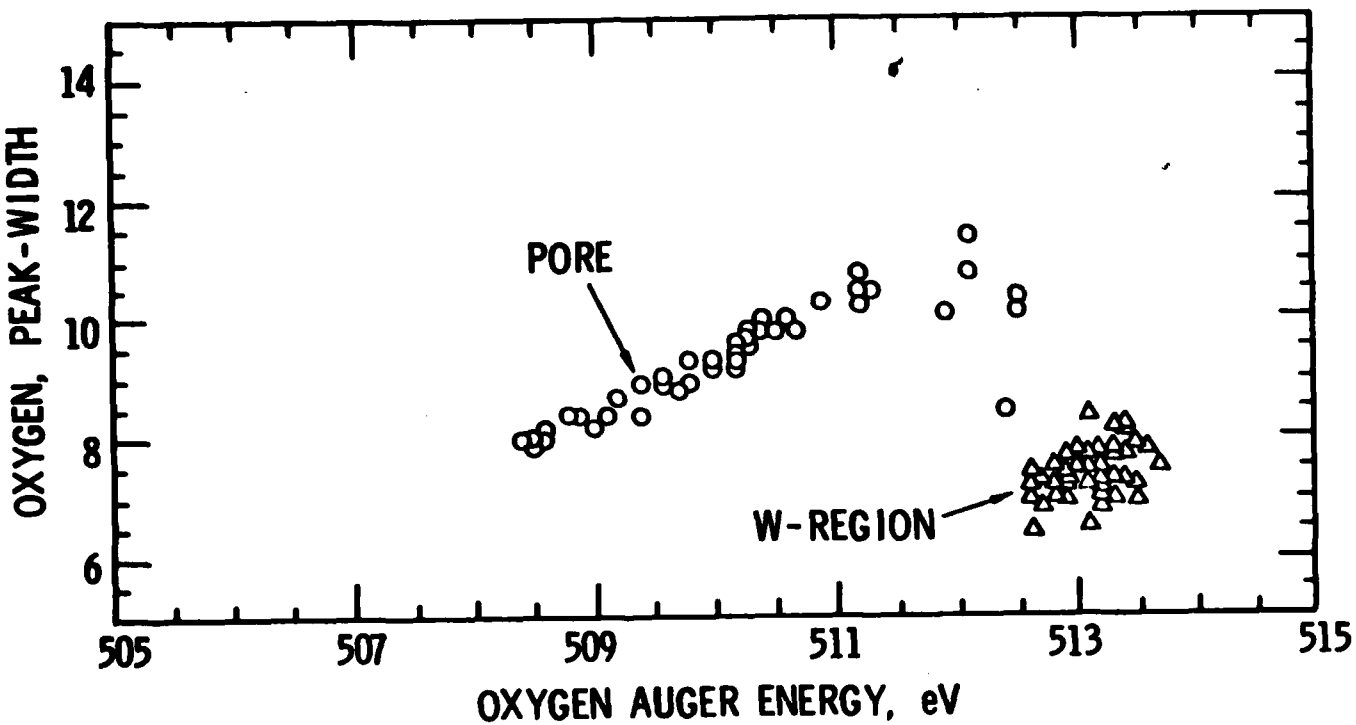

Fig. 1. Plot of Oxygen Peak Width Versus Auger Energy, Taken on Pore and Tungsten Region During Cathode Reactivation. The different oxygen characteristics are used to separate pore-like and tungsten-like regions.

The changes in elemental composition between the new and the 1191°C-aged cathodes are presented in Fig. 2, as a plot of Ba/W ve ratios.[12] The ratio of elements can be used to remove effects cau surface roughness[13] and sample orientation.

The tungsten regions indicate a decrease in barium with age, with a decreased barium supply from the pores. Along with the sur decrease, Fig. 3, plotting O/Ba versus W/Ba shows an increasing as with age of oxygen with tungsten. A Ba-O-(W) surface site model,[6] surface oxygen solely associated with barium sites, would have pre O/Ba ratio to be independent of the W/Ba value. This is most near for the new cathode, but it does not describe the majority of the aged cathodes.

Figure 4 shows the Auger elemental data from the new cathode, into impregnant and tungsten regions by using Fig. 1. The impregn (Fig. 4a) shows two distinct subdivisions. The low W/Ba values co exposed pore ends, or impregnant residues on the surface. The hig ratios correspond to tungsten surface regions with an oxygen signa to the impregnant. These high W/Ba regions in Fig. 4a have a near O/Ba ratio, indicating a barium-oxide composition. Figure 4b, whi elemental composition of the tungsten regions, shows a tungsten ox ture, even on this new cathode. Thus, the separation by using the Auger peak analysis also shows consistent differences in the eleme sition of the microregions.

The results of the aging study (Fig. 3) are superimposed in I show the overall path of the aging process, from an impregnant-lik early life, to a tungsten oxide surface in late life. Two lines w Fig. 5b to outline an oxidized tungsten band in the data. These l redrawn on Figs. 5a and 5c, which show data taken at the lower (~ higher (~1200°C) analysis temperatures. The similarity in the dat Figs. 5a, b, and c indicates that the tungsten regions have a simi composition in the entire range of 1000 to 1200°C. The slopes in

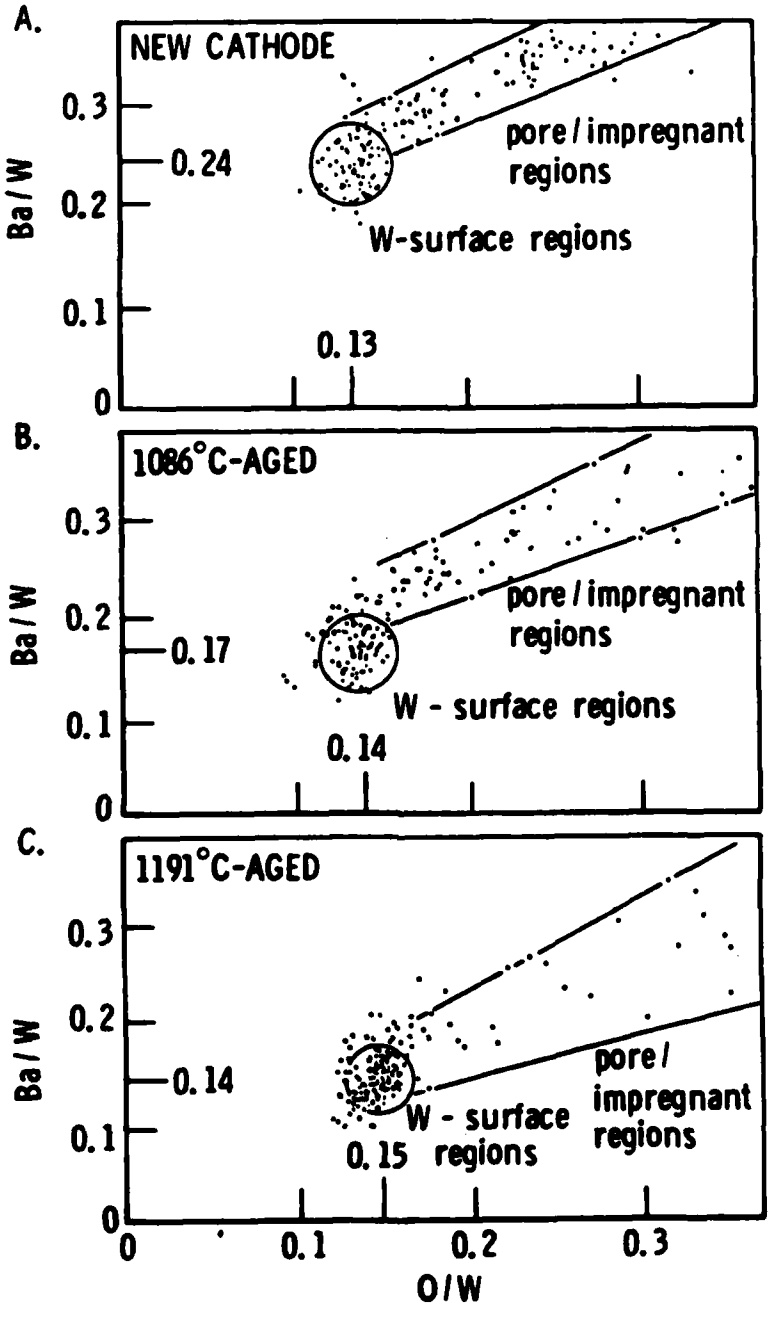

Fig. 2. Comparison of Elemental Composition of New and Aged Cathode Surfaces While Operating Them at 1102 ± 2°C. The boundaries approximately separate pore-and tungsten-type surface regions.

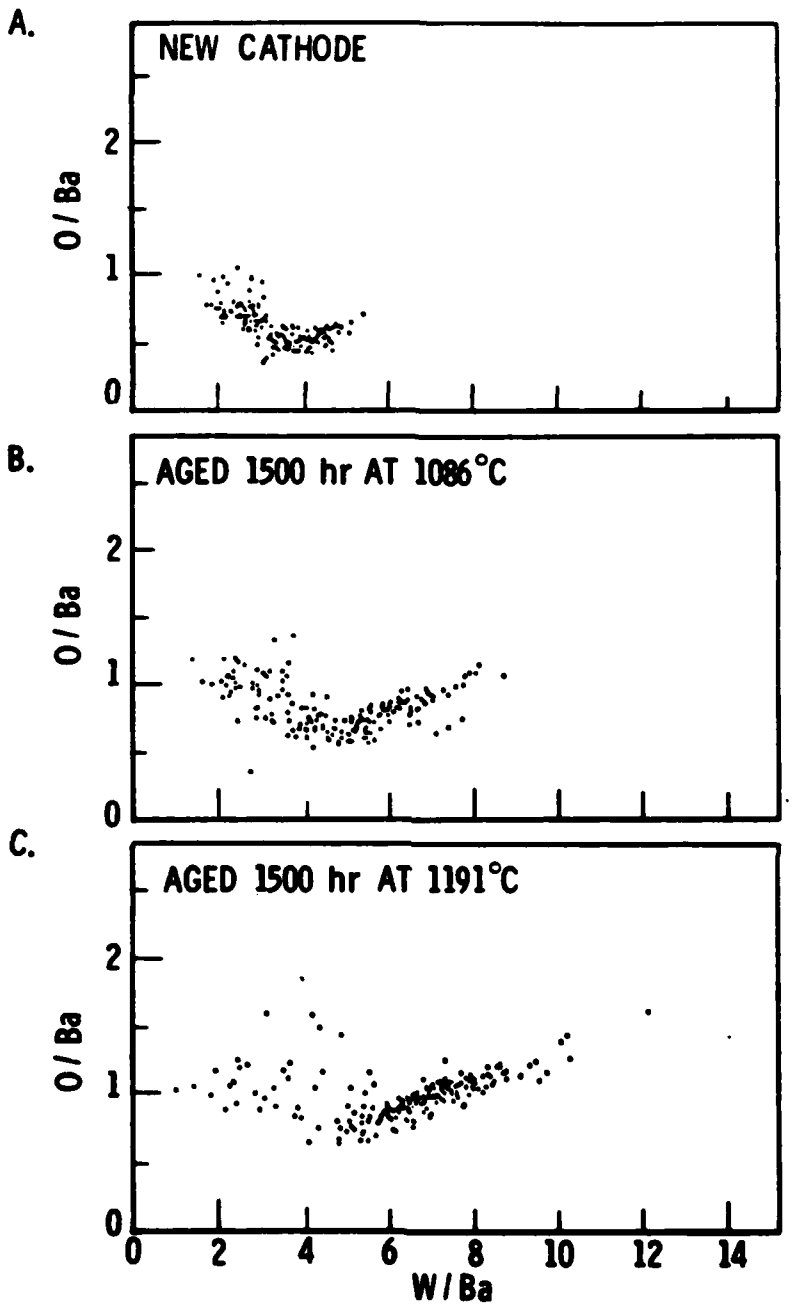

Fig. 3. Oxygen/Barium Versus Tungsten/Barium Elemental Correlation, Showing Development of Surface Tungsten Oxide with Cathode Aging. Data were taken with cathodes at 1102 ± 2°C.

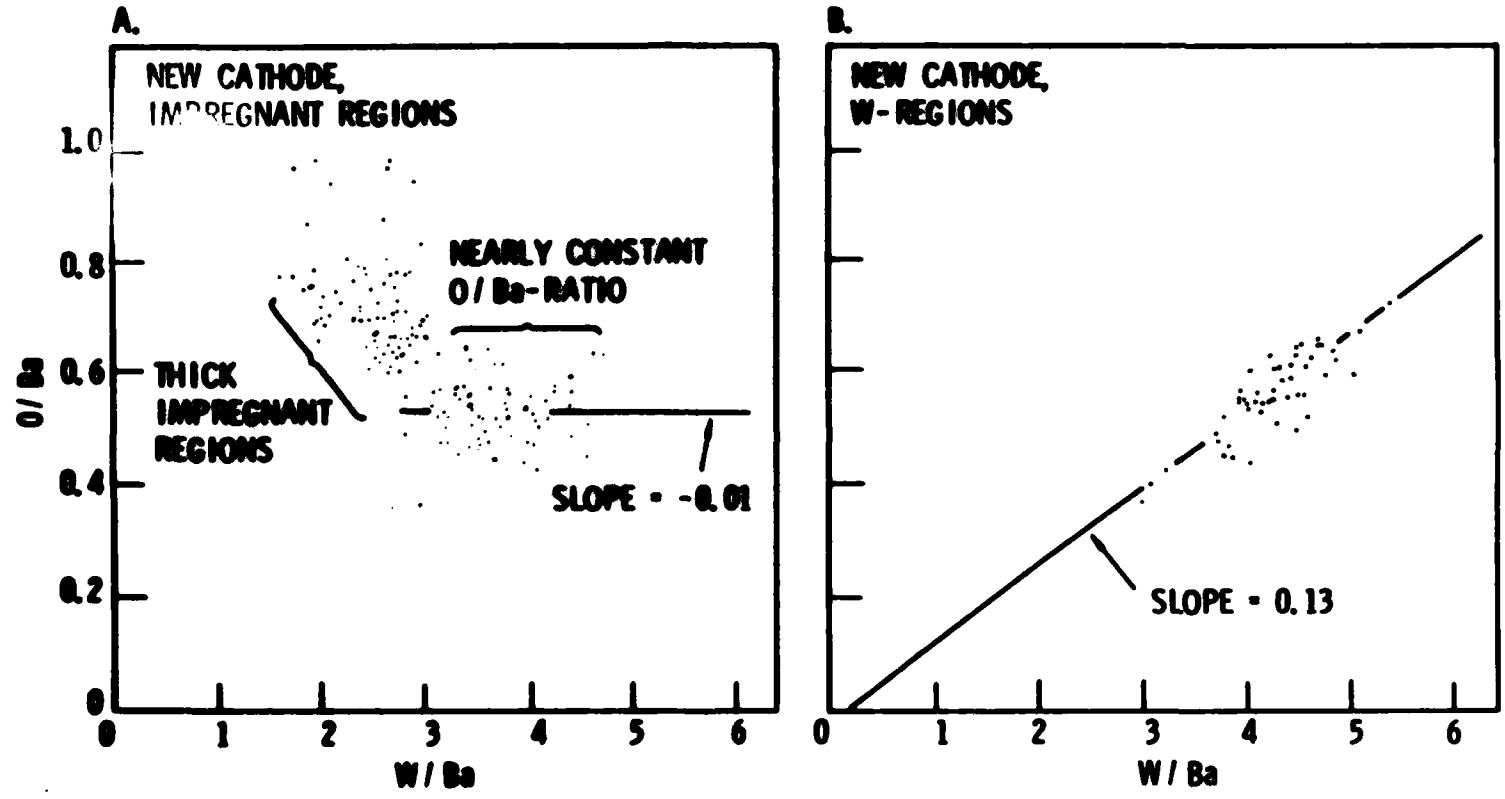

Fig. 4. Separation of Elemental Composition Data, Using Oxygen Peak Characteristics in Fig. 1 to Discriminate Between Pore-Like and Tungsten-Like Regions. This shows that the oxygen signature differentiates between barium oxide and tungsten oxide type regions on the surface. Data were taken at 1101°C.

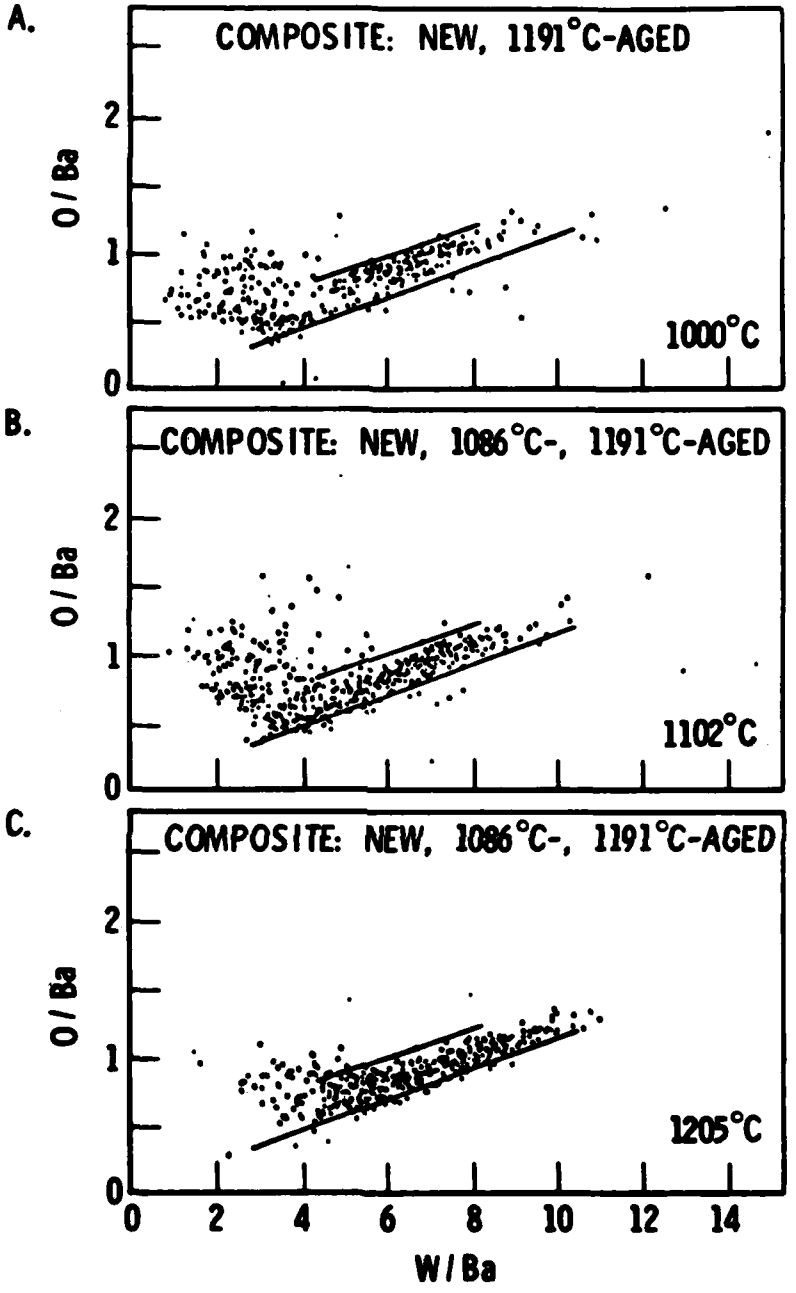

Fig. 5. **New and Aged Cathode Data Superimposed to Give the Overall Path of Cathode Aging Process. The similarity of slope using different cathode operating temperatures indicates that only a small range of tungsten oxide compositions are present on the cathode surfaces.**

and c are nearly identical, indicating that only a narrow range of tungsten oxide stoichiometry is present on the cathode surfaces.

Figure 6 shows the development of this narrow range of tungsten oxide stoichiometry on the surface. These data were taken during the initial heating of the 1296°C-aged cathode. This cathode was not reactivated, and its surface shows the presence of oxygen-rich tungsten oxides. As the temperature was raised, the less stable oxides presumably evaporate resulting in a change of the slope with temperature. After flash-heating for a few minutes at about 1200°C, as shown in Fig. 6c, only the most stable surface tungsten oxide remains. The slope then locks into the oxidized tungsten band of the other aged cathodes.

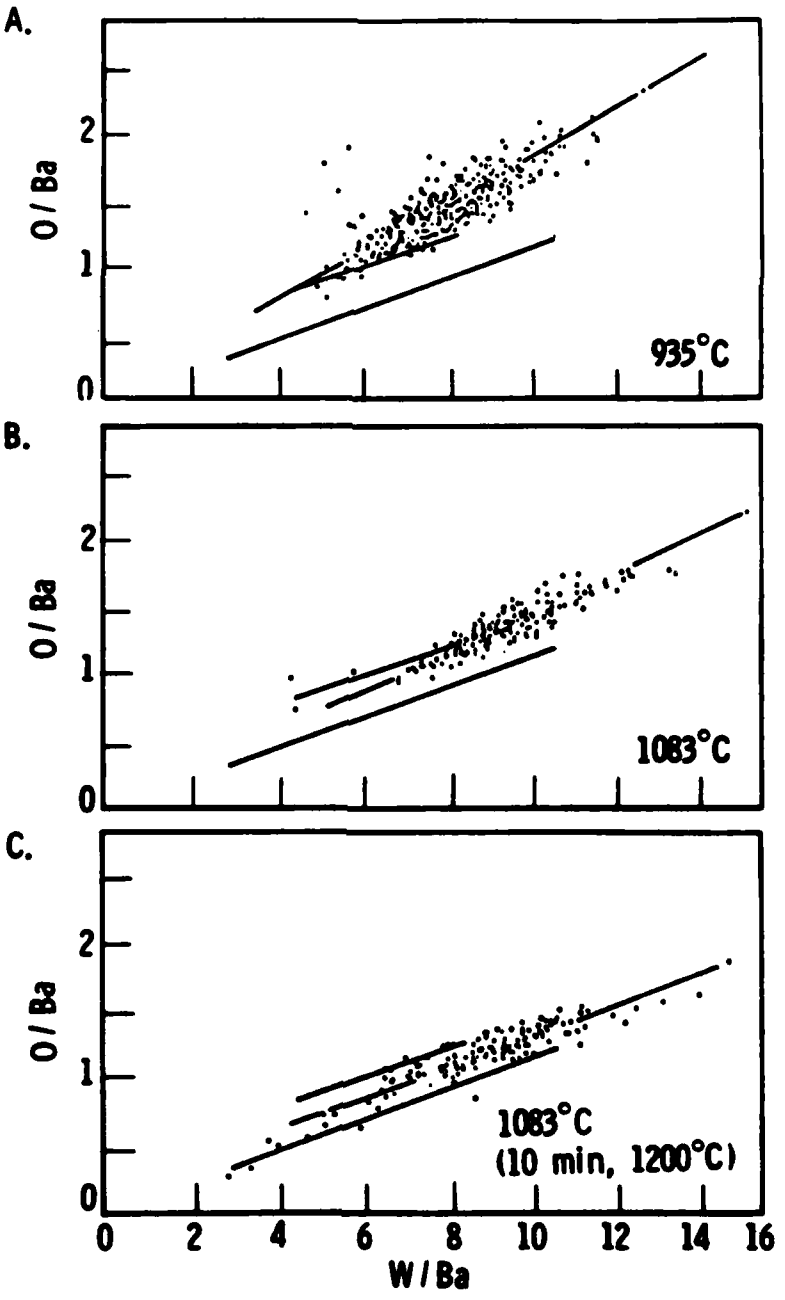

Fig. 6. Surface Tungsten Oxide Development, on 1296°C-Aged Cathode, During First Heating of Cathode After Air Exposure. At low temperatures, oxygen-rich tungsten oxides are present, but the high temperatures of activation (~1200°C) remove all but the most stable oxide species.

IV. CONCLUSIONS

Surface changes occurring with age on a dispenser cathode were examined using a computerized SAM system, and using temperature-accelerated aging for the cathodes. The major surface signatures of aging were a decreased barium coverage and a change in the elements that are associated with oxygen. A corresponding change is seen on the surface in the peak width and position of the oxygen Auger line. With these results, we have been able to construct the following picture of cathode operation, from early life through middle age.

A cathode exposed to atmosphere builds up a surface composed of oxygen-rich tungsten oxides. The high temperature associated with activation (~1200°C) helps to remove these oxygen-rich compounds, resulting in a more uniform surface containing the most stable tungsten oxides. When the cathode is cooled to a given operating temperature, the surface establishes a new equilibrium between supply and evaporation. On a new cathode, this equilibrium point has most of the surface oxygen associated with barium sites. As the cathode ages, the barium supply lessens, and more surface oxygen gradually becomes associated with tungsten sites. This changes the surface from similar to barium oxide on tungsten, to one of an increasing fraction of tungsten oxide with age.

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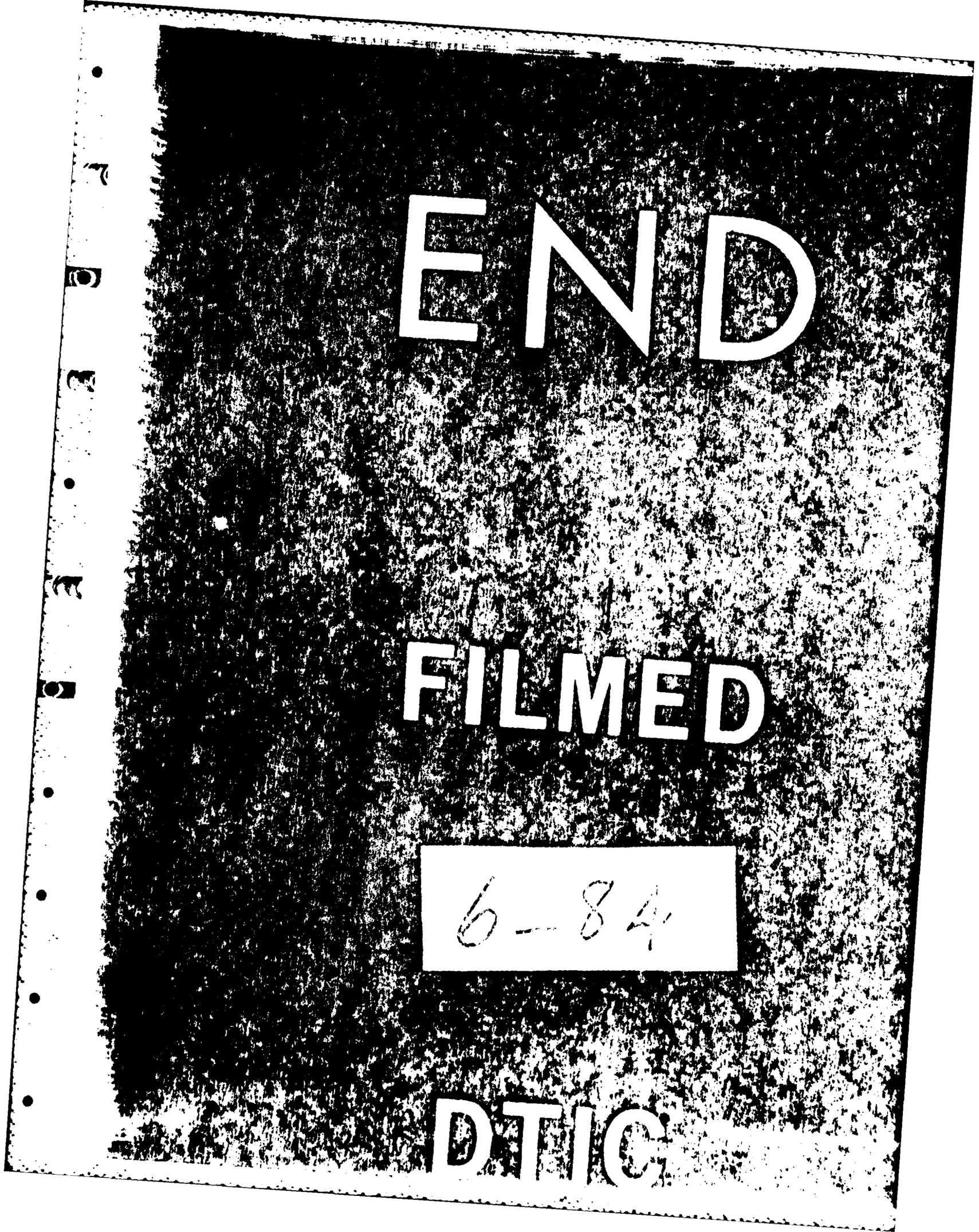
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