



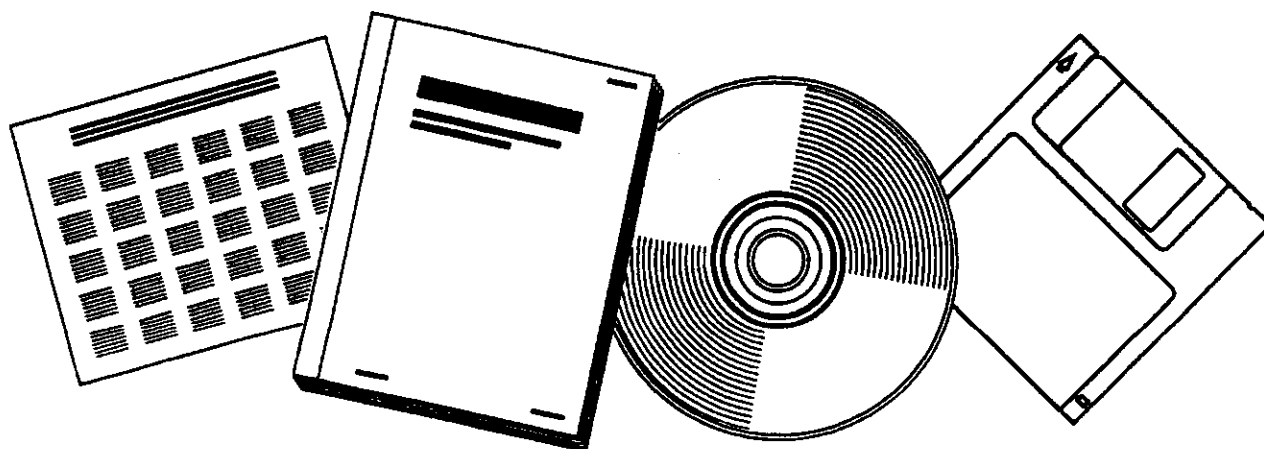
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SPECTROSCOPY OF METAL/TITANIA SYSTEMS

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Spectroscopy of Metal/Titania Systems

by

Y.-M. Sun, D. N. Belton and J. M. White

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Thin film models of catalysts known to show strong metal support interactions were investigated. These consist of vapor-deposited Pt or Rh on TiO ₂ films prepared in ultrahigh vacuum by oxidation of Ti(000±). TDS, AES and static SIMS were used to characterize the surfaces before and after thermal treatment in vacuum and with or without chemisorbed CO. The results indicate that heating in vacuum to near 750K leads to migration of a reduced form of titania to the metal film surface, accompanied by suppression of CO and hydrogen chemisorption. In 1 to 2 ML metal films with no surface titania species, no significant suppression of hydrogen chemisorption is seen, indicating that surface segregation alone is not responsible for the changes in hydrogen chemisorption extent and energetics.		

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We have investigated thin film models of catalysis which are known to show strong metal-support interactions. These models consist of vapor-deposited platinum or rhodium on titanium dioxide films prepared in ultrahigh vacuum by oxidation of Ti(0001). Thermal desorption spectroscopy, Auger electron spectroscopy and static secondary ion mass spectrometry have been used to characterize the surfaces of these films before and after thermal treatment in vacuum and with or without chemisorbed carbon monoxide. The results indicate that heating in vacuum to temperatures near 750 K leads to the migration of a reduced form of titanium to the surface of the metal films. This migration is accompanied by significant suppression of carbon monoxide and hydrogen chemisorption just as is found in powdered oxide-supported transition metal catalysts. Sputtering removes the segregated titanium-oxygen species, and the chemisorption of carbon monoxide is nearly completely recovered. In thin films of metal that are one to two monolayers thick with no surface titanium species, there is no significant suppression of hydrogen chemisorption, indicating that surface segregation is, by itself, not fully responsible for the observed changes in the extent and energetics of hydrogen chemisorption on these surfaces. Clearly, both site-blocking and electronic (bonding) effects play a role in the observed strong metal-support interaction effects.

Recently there has been a great deal of interest in the systems which exhibit strong metal-support interactions (SMSI) (1-12). This activity was stimulated by the work of Tauster et al. (1,2) reported in 1978 which showed that Group VIII transition metals supported on reducible metal oxides were subject to large-scale suppression of chemisorption of hydrogen when the oxides were reduced at high temperatures. Since then there have been a number of papers and conferences on this subject. Proposed explanations include the migration or segregation of oxide species over the transition metal

particles and electronic effects including Pt-titanium bonding and/or charge transfer from the support to the metal.

We have undertaken a series of experiments involving thin film models of such powdered transition metal catalysts (13,14). In this paper we present a brief review of the results we have obtained to date involving platinum and rhodium deposited on thin films of titanium, the latter prepared by oxidation of a titanium single crystal. These systems are prepared and characterized under well-controlled conditions. We have used thermal desorption spectroscopy (TDS), Auger electron spectroscopy (AES) and static secondary ion mass spectrometry (SSIMS). Our results illustrate the power of SSIMS in understanding the processes that take place during thermal treatment of these thin films. Thermal desorption spectroscopy is used to characterize the adsorption and desorption of small molecules, in particular, carbon monoxide. AES confirms the SSIMS results and was used to verify the surface cleanliness of the films as they were prepared.

Experimental

The experiments were conducted in an ultrahigh vacuum chamber equipped with a cylindrical mirror electron energy analyzer, a quadrupole mass spectrometer with a Bessel box energy filter for analysis of secondary ions, and an Ar⁺ gun. A Ti(0001) single crystal was mounted on a liquid nitrogen cooled and resistively heated manipulator assembly. The surfaces were cleaned by argon ion bombardment and annealing cycles. The transition metals were deposited using thermal evaporation sources. During the experiments, pressures were routinely 3×10^{-10} torr.

The titanium-based thin film catalyst models were constructed by first oxidizing the titanium surface in 5×10^{-7} torr of O₂ for approximately 30 minutes at 775 K. This produced an AES lineshape consistent with fully oxidized TiO₂. The metal was then vapor deposited onto the oxide support with the latter held at 130 K. The thickness of the metal overlayer and its cleanliness were verified by AES. After various annealing and adsorption procedures, these thin films were further characterized using SSIMS, AES and TDS. For comparison, some work was done with Pt on Al₂O₃. In this case a Mo foil covered with Al₂O₃ replaced the Ti(0001) substrate.

Results

Figure 1 shows AES data for the oxidized titanium surface before and after deposition of 30 Å of platinum with the substrate held at 130 K. The platinum thickness was calculated from the attenuation of the oxygen AES signal assuming layered growth of the metal. From the spectra it is clear that the platinum was sufficient to completely attenuate the underlying features of the titanium oxide. The spectra of the oxide surface prior to metal deposition is characteristic of fully oxidized titanium. In the region just below 435 eV the lineshape is significantly different for different oxides of titanium (15).

Figure 2 shows the results of heating a model system consisting of a 30 Å platinum film on oxidized titanium. A linear temperature ramp was applied until the foil reached 760 K, after which the temp-

annealing experiment was carried out. Similar experiments were done with Pt on alumina. As indicated in Table 1, annealing to 760 K does not change either the desorption temperature or the area under the carbon monoxide desorption peak.

Similar SSIMS and TDS results were obtained for rhodium on titania and for hydrogen chemisorption on both substrates. In a blank experiment involving no metal overlayer, temperature programming while following the Ti and TiO⁺ SIMS signals (Fig. 4) shows that the titania thin film does not begin to change until the temperature reaches about 760 K, well beyond the 615 K where Ti⁺ was first noted to increase on the systems with thin metal overlayers.

To explore the possibility of electronic interaction between the reduced titanium oxide and Pt, 1 ML of Pt was deposited on both fully oxidized and partially reduced TiO₂. The reduced sample was prepared by Ar⁺ sputtering of the TiO₂ substrate prior to metal deposition, introducing reduced Ti centers at the Pt-Ti interface. H₂ TDS was used as the highest H₂ desorption temperature (370 K) occurs below the temperature regime of encapsulation. For the reduced sample there was a 70% decrease in H₂ chemisorption and a 33 K shift to lower temperatures when the unannealed sample (first H₂ TDS) was compared to the sample annealed at 370 K (second H₂ TDS). No change in the AES was observed after either the first or second TDS, showing that the Pt overlayer does not island or encapsulate. We take these low Pt coverage experiments to indicate an electronic interaction (preferably bond formation, which does not require significant charge transfer) between Pt and reduced Ti species that is activated at about 370 K.

For the fully oxidized sample the results were somewhat different. After annealing at 370 K there was 25% less H₂ adsorption, and the peak split into two peaks, one shifted higher and one lower, each by about 30 K. There were also small changes in the AES Pt/Ti ratio after the first TDS. Since TiO₂ migrates at lower temperatures for reduced titania, as compared to fully oxidized, we do not favor TiO_x migration as the explanation. Rather, we suggest that the changes observed after the 370 K anneal of the oxidized sample are due to small changes in the morphology of the Pt overlayer. These alter the number and kind of exposed Pt sites.

Discussion

From the SIMS, AES and TDS data the following picture emerges. Oxidation of titanium in situ leads to the formation of a film of fully oxidized titania that is thick enough to completely attenuate metal and suboxide contribution to the AES spectra. Heating these overlayers, which are judged to be more than 60 Å thick, results in no detectable changes by SIMS or by AES (not shown) until the temperature exceeds 760 K. Since thermal effects are observed at significantly lower temperature when metal overlayers are present, we conclude that those observations are not due to degradation of the oxide layer via diffusion of oxygen into the bulk metal.

The AES signals observed after deposition of platinum (Fig. 1) indicate that platinum goes down reasonably uniformly under our conditions. This result is confirmed by a more detailed analysis of the attenuation of the oxygen and titanium signals as a function

erature was held constant for approximately 5 minutes and then the sample was cooled. During these temperature changes the Ti⁺ SSIMS signal was followed. Near 615 K the Ti⁺ signal begins to rise sharply. This signal continues to rise during the period when the temperature is held constant at 760 K. Upon cooling there was no tendency for the signal to decrease.

A similar sample was heated as above and then subjected to argon ion sputtering, as indicated in Fig. 3. An argon ion current of 0.3 mA was utilized and the Ti⁺ signal was followed as a function of sputtering time. This figure indicates a sharp decay of the Ti⁺ signal (followed by a relatively wide region where very little signal is noted. Deeper into the sample the Ti⁺ signal rises steadily, maximizes at about 600 seconds and then decays.

On a third sample, thermal desorption of carbon monoxide was carried out before and after annealing to 760 K and after sputtering into the region where the Ti⁺ was a minimum (i.e., about 180 seconds in Fig. 3). The TDS results are summarized in Table 1. The last column gives the integrated peak desorption area for carbon monoxide.

Table 1. CO Thermal Desorption Spectra.

Substrate	Anneal Temp (K)	Desorption Peak (T)	Total Peak Area
TiO ₂	525	400	1.00
TiO ₂	775	355	0.33
TiO ₂ (S)	525	400	0.95
Al ₂ O ₃	525	400	1.00
Al ₂ O ₃	775	400	0.98

(S)-sputtered (see text)

Two CO desorption peaks (400 and 510 K) were observed on the as-deposited Pt layer. However, for a sample annealed to 525 K the intensity of the high temperature CO peak was one third its original value. Thus, after annealing to 525 K, the desorption profile consisted of a large peak at 400 K with a shoulder at 510 K. For this reason we report the peak temperature for the low temperature peak only; however, the 510 K shoulder is included in the reported peak area. Comparison of our data to results from other surfaces shows reasonable agreement. Polycrystalline Pt(415 K, 507 K) (16), Pt(110) (430 K, 530 K) (17) and Pt(111) (420 K, 530 K) (18) all have two desorption peaks in the 400-550 K range. For both titania- and alumina-supported Pt, the areas are normalized to the desorption area after annealing the 30 Å overlayer to 525 K and adsorbing CO to saturation at 130 K. For titania annealed to 775 K the peak desorption temperature is decreased by about 45 K, and the total area is decreased by a factor of 3. After sputtering to the minimum and reannealing to 525 K, the peak temperature returns to 400 K and the total peak area increases to approximately its value before the

of platinum dosing time. From these results (not shown) we conclude that platinum deposition occurs in a very nearly layer-by-layer fashion at 130 K. Annealing these films to 525 K does nothing to the Ti⁺ SMS signal, but at around 600 K Ti⁺ signals begin to appear in the SMS spectra. These rise sharply to a fairly steady value which increases slowly with time. This is a thermally irreversible change since, upon cooling, (Fig. 2) the Ti⁺ signal does not return to a low value. The species formed is largely segregated to the surface as indicated by this sputtering profile of Fig. 3. Sputtering removes this overlayer and leads to metallic platinum. After sputtering through the platinum the oxide region is again reached. After 600 seconds the metal layer is removed, and the Ti⁺ signal begins to drop. This final drop is the result of a decreasing cross-section for ion desorption which accompanies the preferential removal of oxygen.

The observed thermal desorption results are entirely consistent with the picture that emerges from Figs. 2 and 3. Annealing to 525 K (Table I) gives a thermal desorption peak for carbon monoxide like that observed from bulk platinum films. Annealing to 775 K lowers the desorption peak temperature and, more importantly, attenuates sharply the amount of carbon monoxide that will adsorb. Sputtering to the minimum of the Ti⁺ signal followed by an anneal to 525 K and adsorption gives a result that is very much like that observed prior to the high-temperature anneal. From these and other more detailed results involving Auger lineshape studies, we conclude that the species that migrates to the surface of the platinum is probably TiO.

For similar samples on alumina, these effects are not observed, as indicated in Table I. No migration of aluminum or oxygen species is observed in AES, and the capacity of the film to adsorb carbon monoxide is not altered by changing the annealing temperature from 525 to 760 K.

All of these results are consistent with the notion that surface migration of titanium oxide species is an important factor that contributes to the suppression of carbon monoxide chemisorption. Ti-H chemisorption experiments on 1-2 ML of Pt, where no migration is observed, strongly indicate that electronic (bonding) interactions are also occurring. Thus, for the titania system, both electronic interactions and surface site blocking due to titanium oxide species must be considered in interpreting SMSI effects.

Acknowledgments

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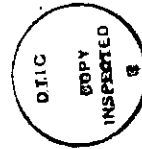


FIGURE CAPTIONS

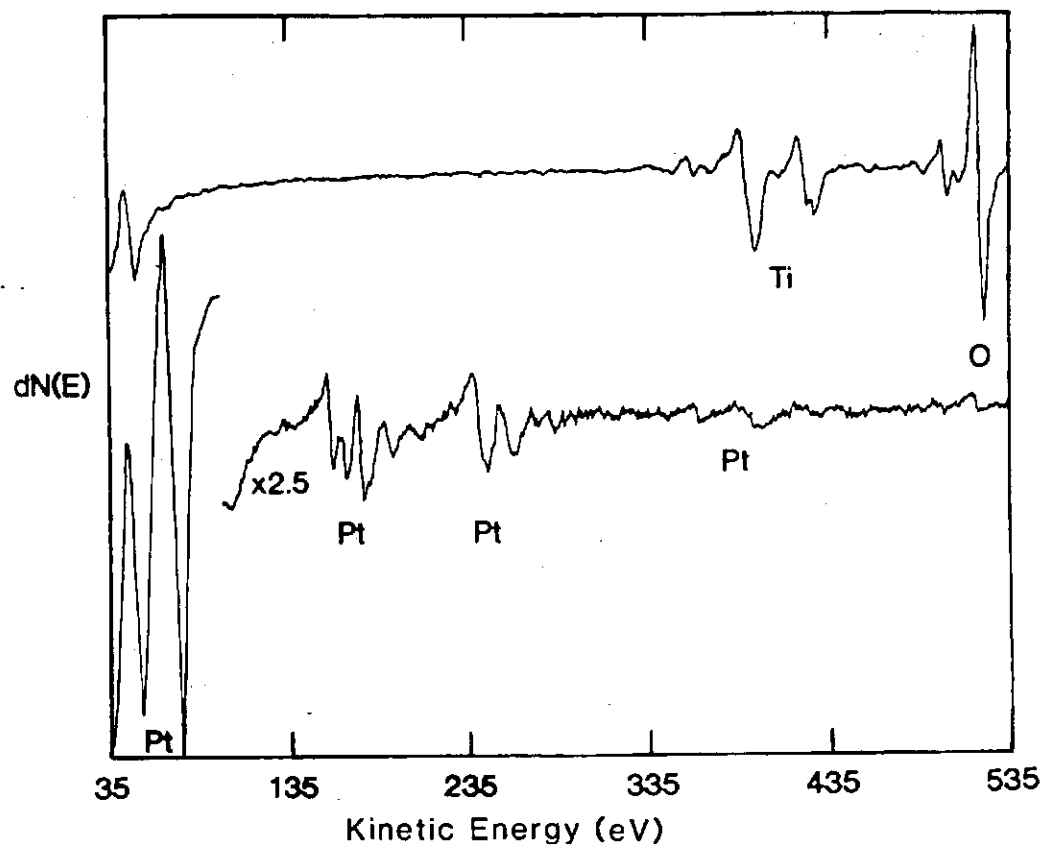
Figure 1. AES spectra of oxidized Ti(0001) (top) and oxidized Ti with 30 Å Pt (bottom).
 Figure 2. TPSSIMS for 30 Å Pt on oxidized Ti(0001).
 Figure 3. Ti⁺ SIMS depth profile of 30 Å Pt on oxidized Ti(0001) annealed to 760 K.
 Figure 4. TPSSIMS of the oxidized Ti(0001) substrate without a metal overlayer.

Figure 1 AES spectra of oxidized Ti(0001) (top) and oxidized Ti with 30 Å Pt (bottom).

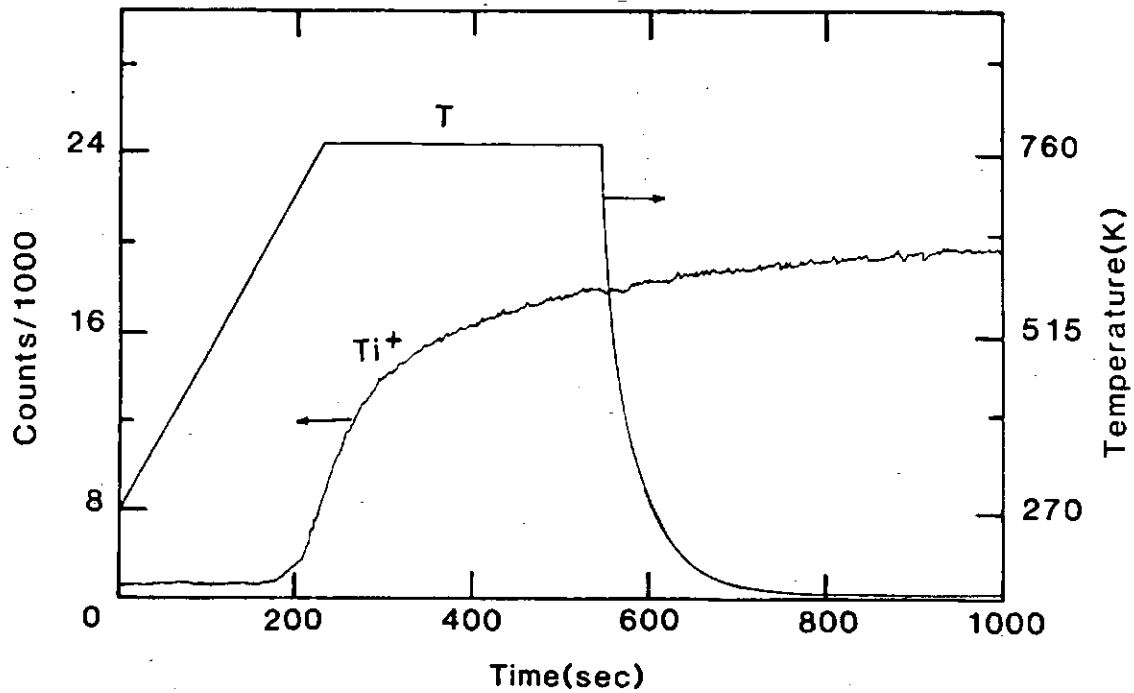
Figure 2 TPSSIMS for 30 Å Pt on oxidized Ti(0001).

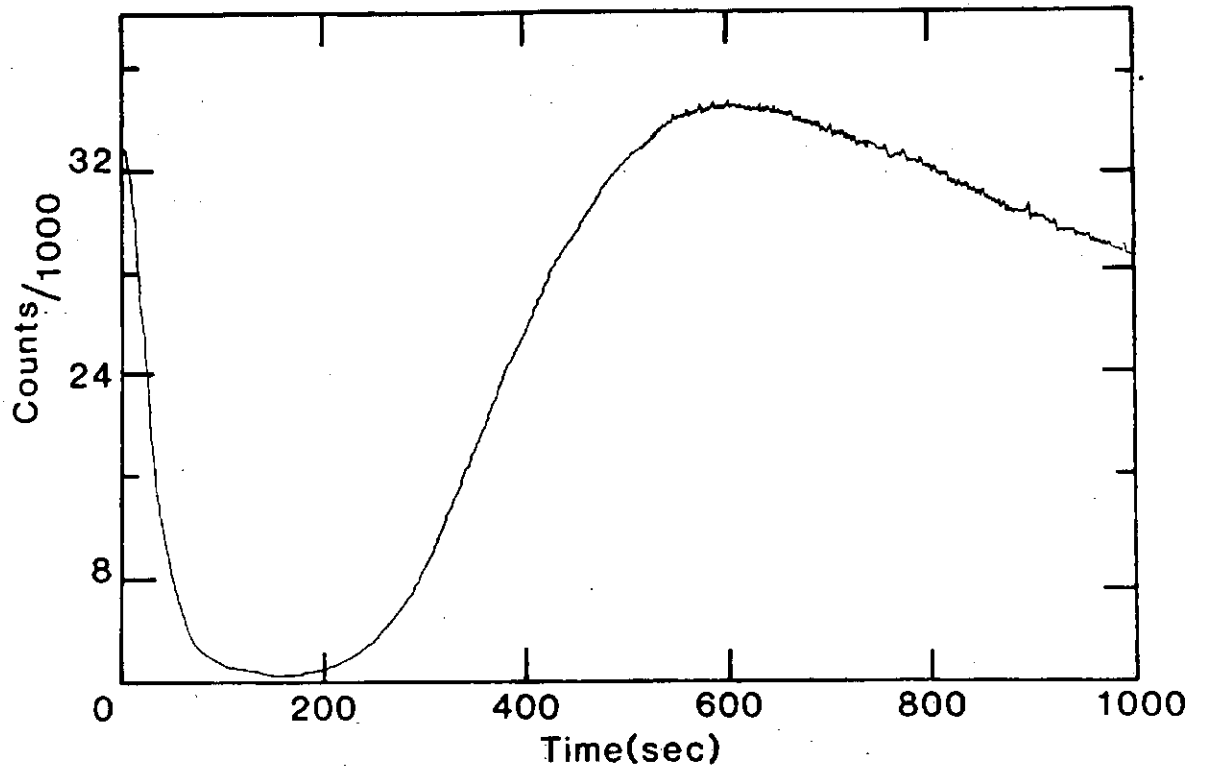
Figure 3 Ti⁺ SIMS depth profile of 30 Å Pt on oxidized Ti(0001) annealed to 760 K.

Figure 4 TPSSIMS of the oxidized Ti(0001) substrate without a metal overlayer.

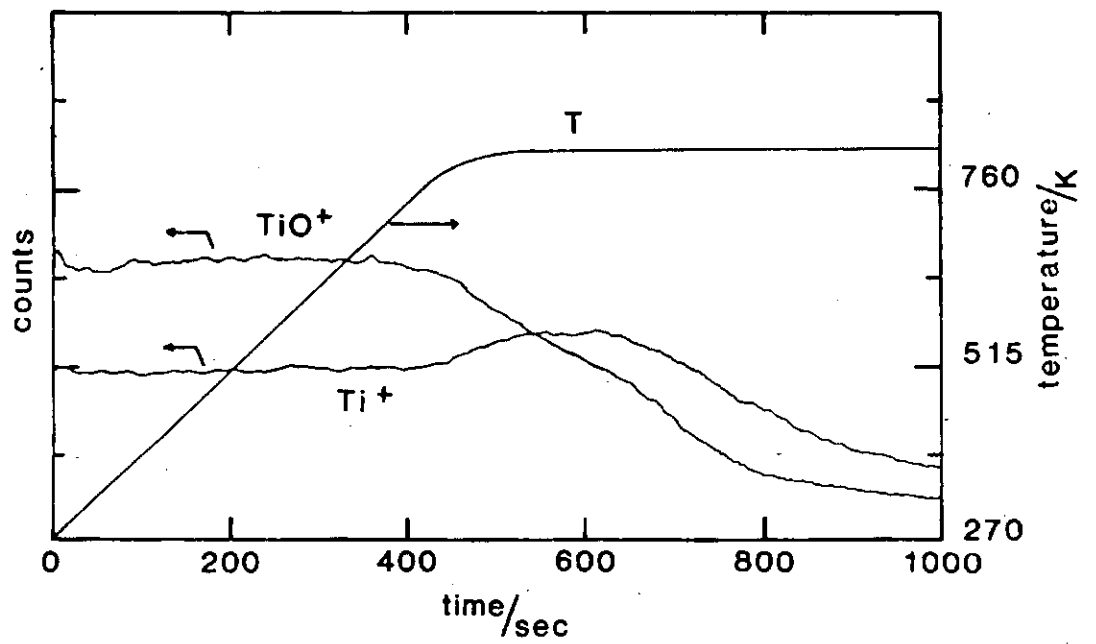


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Figure 1





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Figure 3



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