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INFLUENCE OF HYDROGEN DIFFUSION ON THE RESISTIVITY OF COLD-WORKED NICKEL WIRES

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IT is well known that the result of cold work on a metallic sample is the creation of defects and dislocations in it. The effect of slow-cycle-creep cold work (by winding–unwinding method) on the thermo-emf of nickel wire was reported earlier[1]. Lee and Lee[2] have reported that hydrogen diffused into

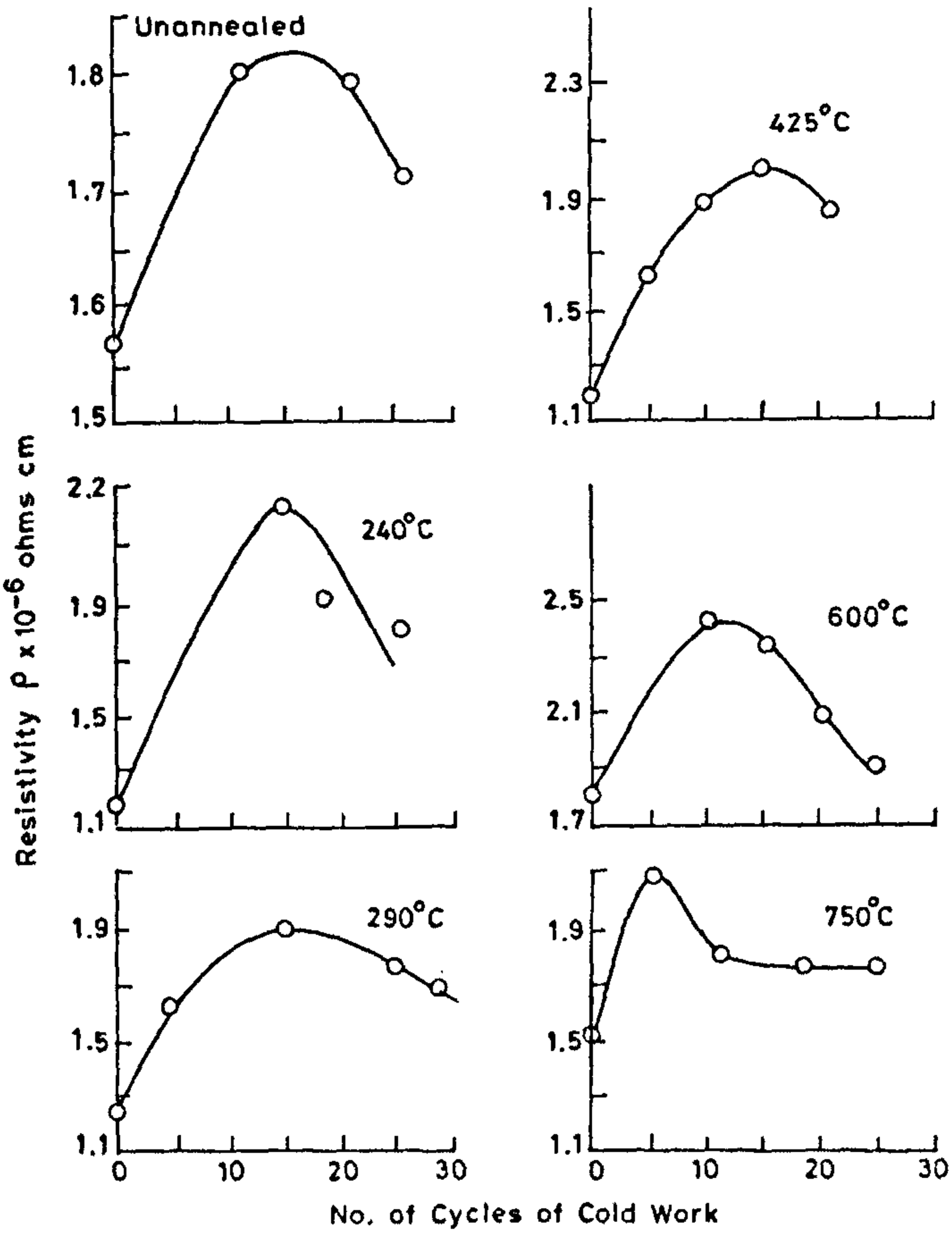

Figure 1. Variation of resistivity with cold work of nickel wires annealed at different temperatures.

nickel gets trapped in the defects. Obviously a cold-worked sample with a larger number of defects will trap a greater amount of diffused hydrogen and show interesting effects in the electrical properties. The present note reports observations on the resistivity of cold-worked wire before and after hydrogen diffusion as a function of the amount of cold work.

Sample preparation and slow-cycle-creep cold working were described earlier in connection with thermoelectric[1] and electrode potential measurements[3]. We had in stock a number of samples pre-annealed at different temperatures and cold-worked by different amounts.

The resistivities of such samples were measured by a ten-wire potentiometer by a comparison method using a sensitive moving-mirror galvanometer for null-point detection.

The samples were subjected to electrolytic cathodic

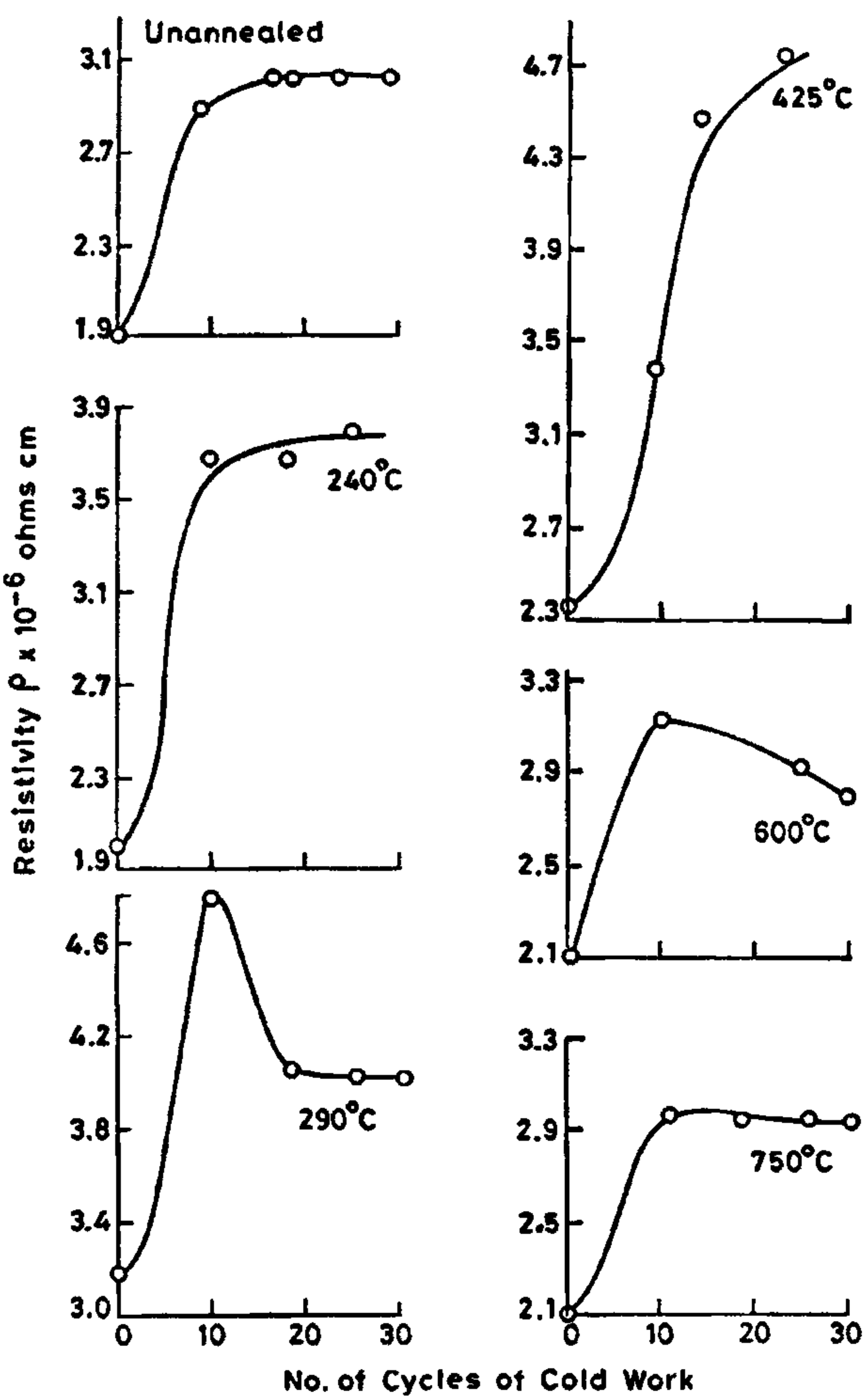

Figure 2. Variation of resistivity with cold work of hydrogenated and outgassed nickel wires annealed at different temperatures.

hydrogen diffusion in the presence of thiourea as an inhibitor; details have been described earlier[4]. During the electrolysis, hydrogen liberated at the nickel wire (cathode) diffuses into it slowly. Such hydrogenated wires, lose the mobile part of hydrogen within 7–8 h while retaining the trapped hydrogen in the lattice. This is indicated by the fact that the outgassed sample does not attain its original values of thermo-emf[4], resistivity[5], rigidity[6], etc. We thus have two sets of samples: (i) differently annealed samples, cold-worked to various degrees (number of cycles of winding–unwinding around a rod under a load of 1 kg), (ii) samples subjected to cathodic hydrogen diffusion and subsequent outgassing of the mobile part of hydrogen.

Figure 1 shows the variation in resistivity with the number of cycles of cold work for samples annealed at different temperatures. Figure 2 shows the resistivity variation after hydrogenation and outgassing. Two salient features are clear from these figures: (i) samples annealed at 290° and 600°C show a different behaviour from the rest (figure 2); 290°C is close to the strain recovery temperature (300°C), while 600°C is the recrystallization temperature, (ii) after hydrogen treatment the samples tend to attain a saturation value of resistivity.

If hydrogen ions are trapped in the lattice vacancies, the medium becomes electrically more homogeneous. As the amount of cold working is increased, the number of defects created increases and so does the amount of hydrogen that can be trapped. The sample shows a tendency to achieve a saturation value in its resistivity.

The effect of annealing on the shape of the *d*-band has been reported[7] earlier on the basis of thermoelectric studies. The process of cold work affects the shape of the *d*-band to the same extent in the different annealed samples. In the present case cold work affected resistivity in different annealed samples to different extents; it is the hydrogen diffusion that tends to restore the disturbed values. Thermoemf is a property dependent on the shape of the *d*-band, but resistivity is dependent on the number and distribution of lattice defects. The effect of hydrogen diffusion seems to be to smoothen out the effect of vacancies which trap hydrogen.

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CHARACTERIZATION OF THE POLYMERIC COMPLEX OF Sn(II) WITH TETRATHIAZYL DIHYDROFLUORIDE BY IR, UV AND EPR SPECTROSCOPY

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SYNTHESIS and investigations of tetrathiazyl dihydrofluoride (TTADHF) and its complexes with Ti(III) and Zr(IV) have been reported[1,2]. The preparation and studies of the complex of $SnCl_2$ with TTADHF are presented here.

TTADHF was prepared by mixing dry H_2F_2 into a benzene solution of S_4N_4 synthesized by Goehring's method[3]. To prepare the complex, DMF solutions of $SnCl_2$ and TTADHF were mixed together in equimolar ratio and refluxed for about 24 h. The brownish-red product formed was separated and washed with DMF till the washings were colourless. It was dried and stored *in vacuo*. For estimations, the complex was analysed qualitatively and quantitatively by atomic absorption spectroscopy and gravimetrically[4]. Molecular weight was determined by viscosity method. IR (KBr Pellet) and UV (reflectance) spectra were obtained on Perkin Elmer 257 and VSU-22 spectrometers respectively. The EPR spectrum was recorded on X-E-4 band EPR spectrometer at 300 K.

The complex is hard. It does not melt but decomposes above 400 C, and is insoluble in benzene and alcohol. The molecular formula was determined from its molecular weight, which was found to be 1646.5. Chemical analysis % found S, 31.05; N,