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WAL TR 830.3/4

# WATERTOWN ARSENAL LABORATORIES

THE ROLE OF DILUTE BINARY TRANSITION ELEMENT  
ADDITIONS ON THE RECRYSTALLIZATION OF COLUMBIUM

TECHNICAL REPORT NO. WAL TR 830.3/4

BY

ERNEST P. ABRAHAMSON, II

JULY 1961

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GENERAL MATERIALS PROBLEMS, RESEARCH AND INVESTIGATION

WATERTOWN ARSENAL  
WATERTOWN 72, MASS.

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Transition elements  
Columbium

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TITLE

THE ROLE OF DILUTE BINARY TRANSITION ELEMENT ADDITIONS  
ON THE RECRYSTALLIZATION OF COLUMBIUM

ABSTRACT

The effect of transition element binary solid solution additions upon the recrystallization temperature of columbium has been investigated. The elements Mn, Fe, Co, Ni, W, Re, and Os lower the recrystallization temperature, while Ti, V, Cr, Zr, Mo, Ru, Rh, Pd, Hf, Ta, Nb, Ir, and Pt raise it. A correlation is noted between the rate of change of recrystallization temperature with atomic percent solute and the free atom electron configuration of the solute element.



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## INTRODUCTION

The work of Abrahamson and Grant<sup>1</sup> has indicated a correlation between the free atom ground state electron configuration and the change in brittle-to-ductile transition temperature per atomic percent solute in chromium-base alloys. A similar correlation was observed for the change in the recrystallization temperature of iron-base alloys by Abrahamson and Blakeney<sup>2</sup> and Abrahamson<sup>3</sup>. It was also observed that the limit of rapid change in recrystallization temperature also correlated with the electronic configuration of the free solute atom. A further study by Abrahamson<sup>4</sup> established the generality of the phenomenon by yielding similar results for vanadium-base dilute alloys. It has been inferred that a periodicity might be expected with changes in the free atom electron configuration of the solvent element.

This study is the third set of transitional base metal systems to be studied. It is the object of this and further studies on transitional metal systems to detect any systematic variations in the observed correlation which might contribute to the understanding of the electron interactions which are evidently exhibiting themselves.

## PROCEDURE

All alloys were made using 99.9 percent Cb with 0.030 Ta, 0.016 O, 0.0025 H, 0.003 Mo, 0.021 Fe, 0.005 C and 0.002 N. The solute elements were 99.9+ percent pure. According to the published binary phase diagrams<sup>5</sup> and metallographic examinations at 750X, all additions were in solid solution.

The alloys were arc melted and remelted six times in the form of cubic 400-g buttons under an argon atmosphere. They were then hot forged at 1200 C to 0.6 inch diameter, annealed for three hours at 1200 C under argon and furnace cooled. The specimens were then machined to 0.400 inch diameter. Grain size was checked and found to remain essentially constant at  $150 \pm 30$  grains per sq mm. The specimens were then swaged to 0.187 inch diameter, yielding  $46 \pm 1$  percent cold work. All alloys were chemically analyzed for the major solute. Analyses of random samples indicated that the impurity content remained at the values of the starting material. Tungsten contamination was held to 0.007 percent.

The swaged rod was cut into 8.25 inch lengths and heat treated in a gradient furnace for one hour. The gradient was 650 to 1200 C over an 8-inch length, recorded by twelve thermocouples, and controlled to  $\pm 3$  C at the hot end.

The recrystallization temperature was determined metallographically as that temperature where the first recrystallized grain appears at a constant magnification, 200X. Specimens were repeated and the agreement was generally found to be  $\pm 2$  C.

## RESULTS

Five different pure columbium rods were tested and the recrystallization temperature was found to be  $990 \pm 1$  C. Figures 1 through 3 show the effect of the transition elements on the recrystallization of columbium. The elements Mn, Fe, Co, Ni, W, Re, and Os lower the recrystallization temperature, while Ti, V, Cr, Zr, Mo, Ru, Rh, Pd, Hf, Ta, W, Ir, and Pt raise it. It should be noted that where breaks from linearity occur, the solubility limits have not been reached.

If one considers the absolute slope of the curves in Figures 1 through 3, a definite periodicity is observed. A plot of the slope versus the free atom ground state outer d shell electron configuration of the solute demonstrates this periodicity, cf. Figure 4.

## DISCUSSION

As shown previously in vanadium- and iron-base recrystallization<sup>2,3,4</sup> and chromium-base brittle-to-ductile transition studies<sup>1</sup>, an electron configuration correlation is present. The form of the columbium plot is the same as the brittle-to-ductile transition temperature plot for chromium. The plot can best be described as a series of V's with their apexes displaced by one d shell electron. This differs from the iron recrystallization data where the apexes are found at the same number of d shell electrons. One fact in common to all of the electron configuration work accomplished thus far is that the apex for the  $s = 2$  electrons V occurs consistently at those elements having one outer d electron more than the free atom configuration of the solvent.

Vanadium and columbium occur in the same group in the periodic table, however, the vanadium-base recrystallization correlation results in an inverted V, while the columbium correlation results in a normal V. It will be necessary to investigate lower d shell configuration transition element solvents, such as titanium and zirconium to note whether the reversal of the V-shaped curve can be attributed to the d shell configuration of the solvent.

It will be noted that breaks in the curves are present in Figures 1 through 3 as in the other systems noted. Due to the lack of sufficient data it was impossible to accurately fix the limit of linearity of all systems.

As in the case of chromium and vanadium, initial negative changes in recrystallization temperature are noted. The negative changes are limited to a consecutive series of elements on the  $s = 2$  electrons curve.



## CONCLUSIONS

1. The elements Mn, Fe, Co, Ni, W, Re, and Os lower the recrystallization temperature of columbium while the other transition elements raise it.

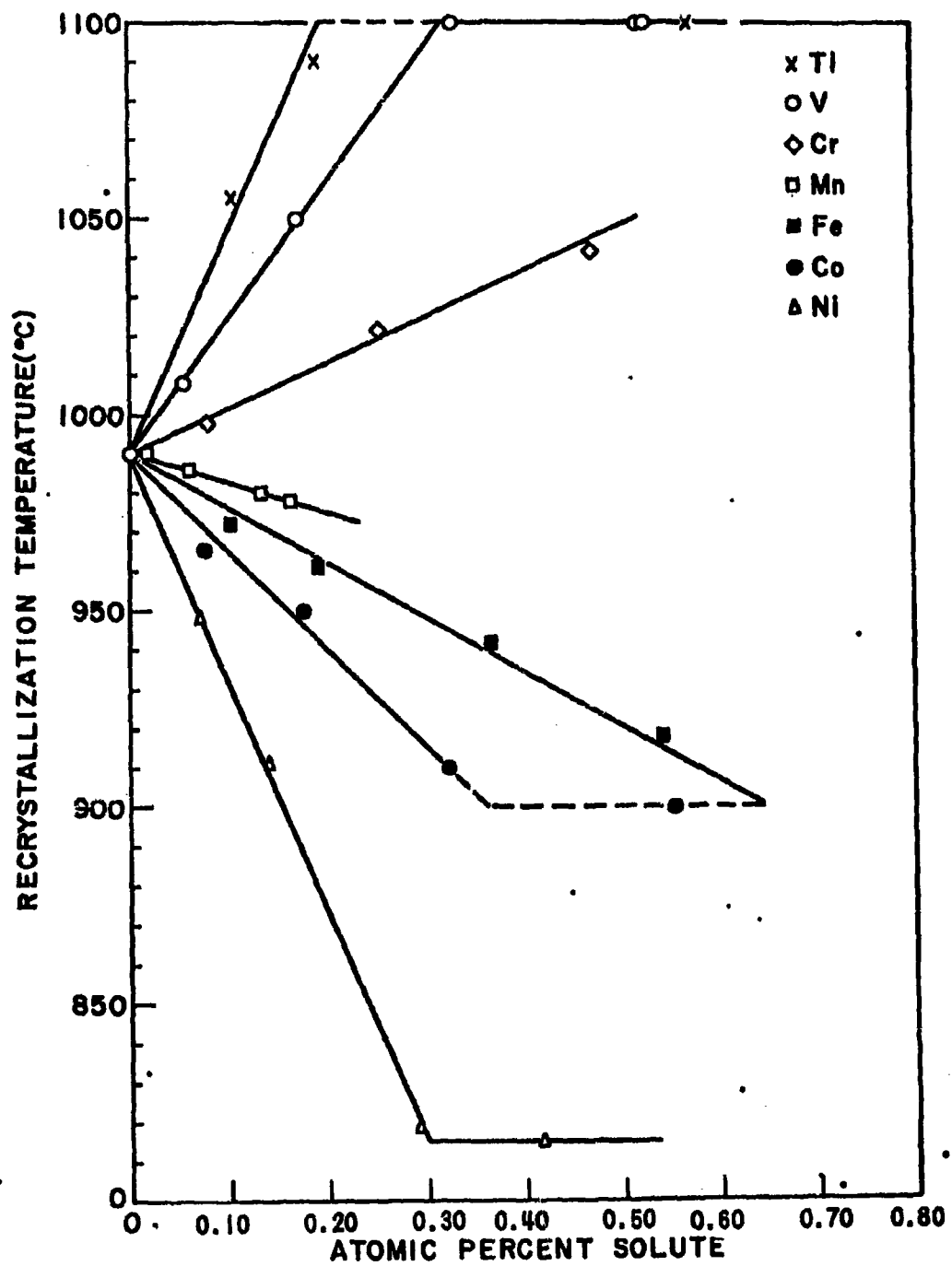
2. A correlation between the logarithm of the rate of change of recrystallization temperature per atomic percent solute and the free atom ground state electron configuration of the solute is observed. This correlation is similar to others carried out on Fe- and V-base materials and for brittle-to-ductile transition temperature in Cr-base alloys.

3. The apexes for the  $s = 2$  curves on all systems studied occur at those elements having one outer d electron more than the free atom configuration of the solvent.

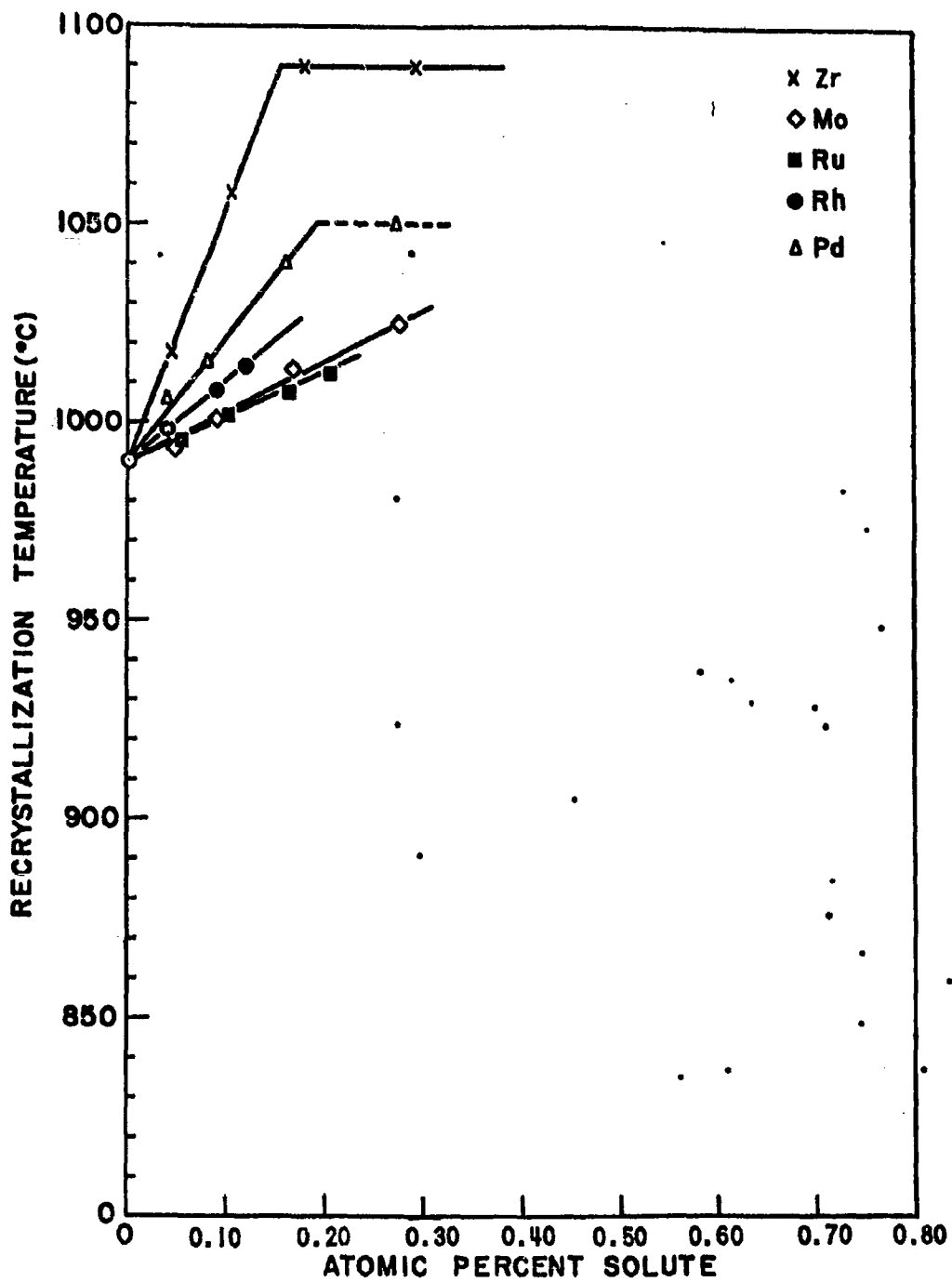
## ACKNOWLEDGMENTS

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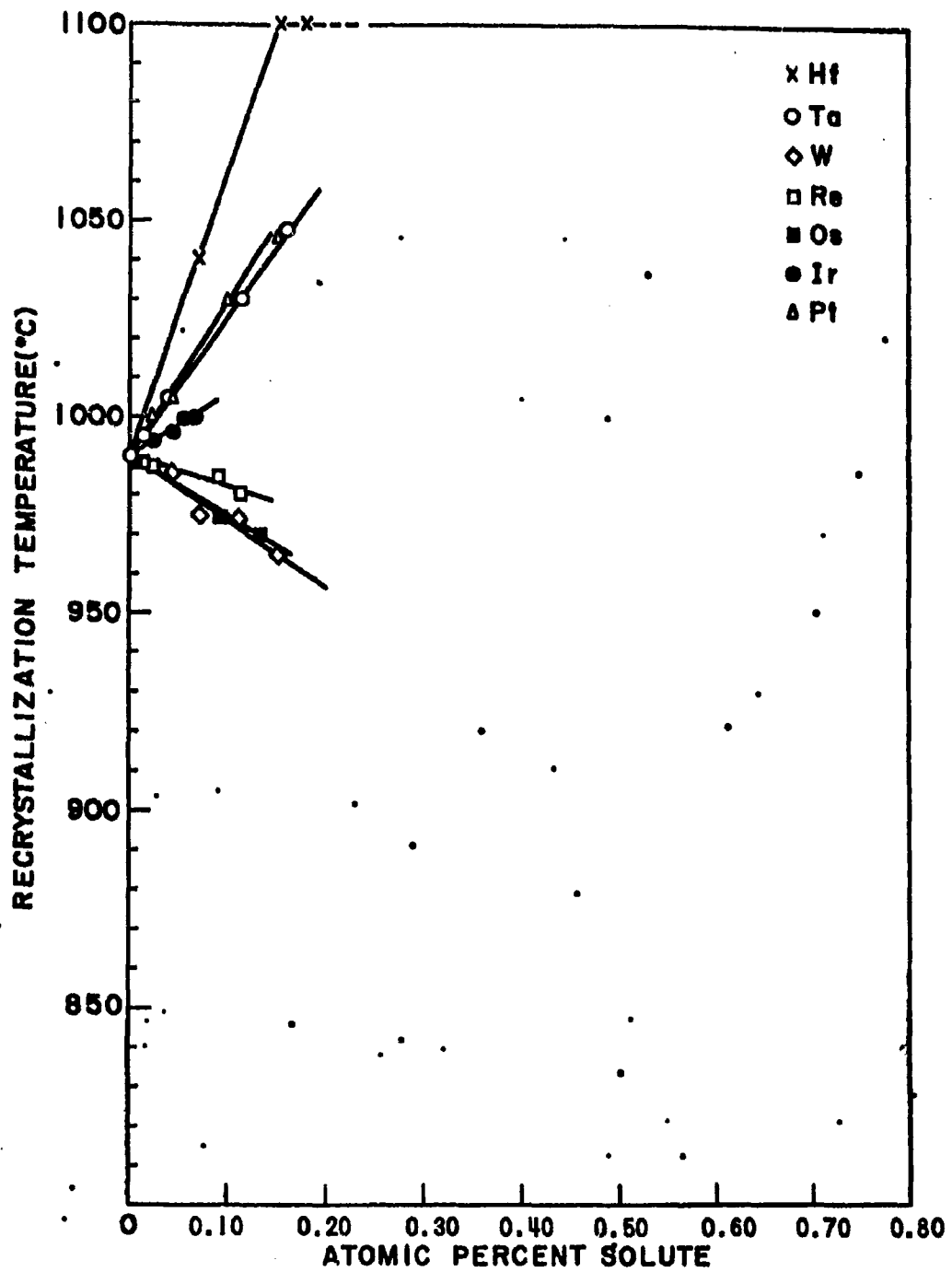
He further wishes to thank Mr. J. A. Alexander and Dr. F. Rhines for their many helpful discussions of the problems.



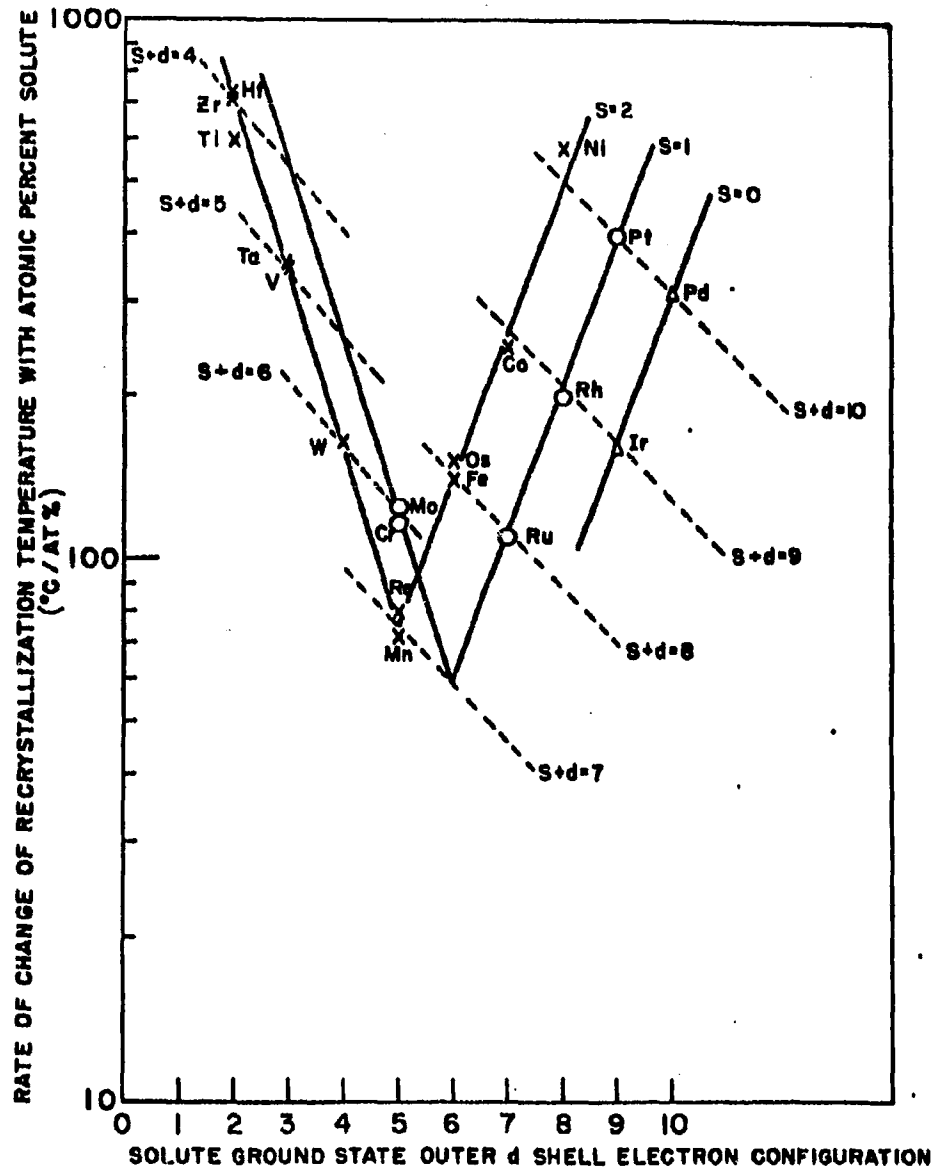
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RECRYSTALLIZATION TEMPERATURE VERSUS ATOMIC PERCENT SOLUTE FOR THE SECOND TRANSITION SERIES.



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RATE OF CHANGE OF RECRYSTALLIZATION TEMPERATURE WITH ATOMIC PERCENT SOLUTE VS GROUND STATE OUTER ELECTRON CONFIGURATION OF SOLUTE.

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