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Drucilla G. Davis
(Ph.D. thesis)

August 1981

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The Hexagonal Close Packed Phase
Stability in the Cobalt-Molybdenum system:
Further Confirmation of the
Brewer-Engel Theory

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ABSTRACT

Solid state catalysis and a thermal gradient furnace technique have been used to investigate the sluggish low-temperature hexagonal close packed to cubic close packed phase transformation in the Molybdenum-Cobalt system. The addition of molybdenum stabilizes the hexagonal phase of cobalt to a higher temperature as predicted by the Brewer-Engel correlation for bonding in metals. The maximum temperature at which this hexagonal phase exists is 849°C.

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract Number W-7405-ENG-48.
To George and Dorothy
Acknowledgements

I want to acknowledge and heartily thank Professor Leo Brewer for his sage counsel and limitless support. I wish that I possessed the ability to express with pen and paper the depth of my respect for him.

I also wish to thank Dr. Gary Knutsen, Dr. David Meschi and Professor Alan Searcy for innumerable helpful suggestions and discussions.

While the members of the entire MMRD technical support staff have helped in countless ways, there are four special people who have patiently and unselfishly contributed to my education. They are Glenn Baum, John Holthuis, Herb Riebe and Jim Severns.

This work was supported by the Director, Office Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract Number W-7405-ENG-48.
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Applications of the Brewer-Engel Correlation to the Cobalt-Molybdenum System

Brewer has extensively developed the Engel theory of transition metal bonding and has applied it to the prediction of phase diagrams. The reader is referred elsewhere to several comprehensive discussions of the Brewer-Engel theory (1,2). The results will be presented below.

Bonding in transition metals arises from the contributions of electrons in two different principal quantum shells. The d-electrons are in inner orbitals which are relatively localized and they participate primarily in bonding between nearest neighbors. The s and p orbitals of the outer electronic shell are well extended and are responsible for fixing the long range order and crystal structure of the metal. The recognition by Engel and Brewer of this distinction between inner and outer shell electrons allows the Hume-Rothery rules (3,4) for structures of metallic phases to be extended to the transition metals.

The Brewer-Engel correlation is applicable to pure transition metals as well as to transition metals alloyed with either other transition or non-transition metals. It also successfully predicts the role played by non-metallic solutes. The theory correlates structure with the average number of outer s and p electrons. Electronic configurations with 1.5 or fewer s,p electrons per atom would result in the
body-centered cubic (bcc) structure; 1.7 to 2.1 s,p electrons per atom would yield the hexagonal close packed (hcp) structure and more than 2.5 s,p electrons would yield the cubic close packed (ccp) structure.

As an example, for cobalt the electronic configuration of the ccp phase is given as $3d^{6.5}4s^{1}4p^{1.5}$ while the configuration of the hcp phase is given as $3d^{7.2}4s^{1}4p^{0.8}$ (5). When these two structures are in equilibrium at the transformation temperature, one can predict the effect of various solutes upon their relative stabilities by use of valence bond models used successfully by Pauling (6). At equilibrium, the ccp phase will correspond to $d^{6.5}s^{1}p^{1.5}$ which is the lowest s,p concentration for the ccp range while the hcp phase corresponds to $d^{6.9}s^{1}p^{1.1}$ which is the highest s,p concentration for the hcp range. A large fraction of the d-electrons of Co are either non-bonding pairs or are non-bonding magnetic electrons in highly localized orbitals. To a first approximation, one can predict the effect of transition metal solutes by whether or not they can alter the average s,p electron concentration beyond the 2.1 electron per atom stability limit of the hcp structure or below the 2.5 electron per atom lower limit for the ccp structure.

On this basis, the bcc metals of groups I through VI with electronic configurations of $d^{n-1}s^{1}$ will stabilize hcp Co relative to fcc Co, where n is the total number of valence electrons. Likewise, hcp Tc, Re, Ru and Os with
$d_{n-2}s^1p^1$ electronic configurations will stabilize hcp Co relative to fcc Co. The addition of fcc metals with electronic configurations of $d_{n-2.5}s^1p^1.5$ such as Mn, Fe, Rh, Ir, Ni, Pd, Pt, Cu, Ag and Au will all increase the average s,p electron per atom ratio above the hcp limit of 2.1. This destabilizes the hcp structure of Co relative to the ccp structure.

The differences in internal pressures and in atomic sizes are important factors in determining the absolute solubilities. For hcp Tc, Re, Ru, Os and Co with $d_{n-2}s^1p^1$ electronic configurations these differences are small enough so that the metals form miscible solid solutions.

The bcc metal solutes such as Cr and W with $d^5s^1$ electronic configurations can attain maximum concentrations of 40 at% and 25 at% respectively in hcp Co for similar reasons. Likewise, the cubic Fe, Rh, Ir, Ni, Pd and Pt are miscible with cubic Co. The internal pressure solubility parameter of Co differs enough from those of Mn, Cu, Ag and Au so that these fcc phases are not miscible. These elements with $d_{n-2.5}s^1p^1.5$ configurations do favor ccp Co over hcp Co, however, and their solubilities at a given temperature are larger in ccp Co than in hcp Co. Table 1 lists the internal pressures of these elements (2).

It has been two decades since the predictions of the Brewer-Engel theory were put forth. Considerable new data have accumulated and the agreement has been in the 95-99%
range for the various types of predictions arising from the theory.

The currently accepted Co-Mo phase diagram contradicts the prediction that the addition of bcc Mo with $d^5s^1$ electronic configuration to Co should stabilize the hcp phase relative to the ccp phase. Therefore it is of interest to reexamine this system more carefully.
The Cobalt-Molybdenum System

The currently accepted phase diagram of Co-Mo (7,8) does not show extension of the hcp α-Co solid solution phase upon addition of Mo. See Figure 1. In contrast to this, Brewer et al. present the phase diagram shown in Figure 2. In their report they have extensively used the regular solution theory and the predictions arising from the Brewer-Engel correlation. In the latter report, two hexagonal close packed phase regions are presented as well established. They are the ordered K-Co₃Mo (hP8) phase of the Ni₃Sn structure which is stable up to 1025 ± 5°C and the θ-Co₉Mo₂ (hP2) isolated phase region which is stable between 1018 ± 5°C and 1200 ± 10°C and between 17 and 19 at% Mo. While a number of experimental studies have been reported, dispute remains concerning the relative stabilities of the hcp α-Co (hP2) and ccp β-Co (cF4) solid solution phases upon addition of Mo.

Hultgren et al. (7) have reviewed the observations of the sluggish hcp α-Co to ccp β-Co transformation and assign a value of 427°C ± 40°C to the transformation temperature. More recent work has confirmed their value (10,11).

Work in the 1920's and 1930's indicated that 6 at% Mo stabilizes the hcp α-Co phase to a temperature between 620°C and 800°C and that additional Mo extends the hcp α-Co phase to a maximum temperature between 915°C and 1140°C (12-14). Sykes and Graff (15) contradicted these results and
suggested that the α-Co phase is an isolated region at 17 at% Mo between 1020 and 1200°C. In 1961, Bibring and Graf (16) reported that hcp α-Co in equilibrium with κ-Co₃Mo extends up to 700°C. Unaware of this report, Quinn and Hume-Rothery (17) did the definitive study on the system in 1963. They studied the system between 800°C and 2300°C and were above the maximum equilibrium temperature for hcp α-Co reported by Bibring and Graf. Quinn and Hume-Rothery confirmed the observations of Sykes and Graff and presented a phase diagram showing the κ-Co₃Mo and θ-Co₉Mo₂ phases but no extension of the α-Co phase upon addition of Mo. This diagram has generally been accepted as conclusive.

In 1970, Krajewski et al. (18) presented data which indicate that the α-Co transformation temperature is raised upon addition of Mo. Heijwegen and Rieck (19), using diffusion couples, have seen that α-Co extends to 1000°C and 16 at% Mo yet is still separated from the θ-Co₉Mo₂ boundary at 1020°C and 18.6 at% Mo. However, shortly after publication of the diffusion couple experiments, Gust et al. (20) reported that only a two phase mixture of ccp β-Co and κ-Co₃Mo exists between 700°C and 950°C for 7-10 at% Mo.

Peritectoid reactions at low temperatures pose a general problem. If the system is studied at a temperature much below the peritectoid temperature, the diffusion rates are too slow to yield equilibration in a reasonable amount of time. If the temperature is above the peritectoid, the
phase is unstable. It is difficult, by trial and error, to find the narrow temperature range below the peritectoid temperature where the desired phase is stable and the equilibration rates are at their maximum.

To obtain a definitive answer for the Mo-Co system, two techniques were employed. A gradient furnace technique (21) was used to determine the maximum temperature of the hcp $\alpha$-Co to ccp $\beta$-Co transformation. Once this temperature was determined, the composition range of the $\alpha$-Co phase was examined using Pb to catalyze the sluggish reaction. A similar technique was used by Brewer and Kane (22) to equilibrate arsenic solid and vapor and by Knutsen, Searcy and Beruto (23) to equilibrate calcium carbonate and calcium oxide.
II. EXPERIMENTAL

Apparatus

The gradient furnace consists of a 1.5" o.d., 1.25" i.d., 36" long mullite tube wound differentially with 18 gauge (0.040" diameter) Kanthal wire. The lower temperature zone has the wire wound 3 turns/inch for 7"; the middle zone has 6 turns/inch for 8" and the high temperature zone has 16 turns/inch for 10". A 2" region of varying wire spacing separates the zones. The windings were coated with an alumina/borosilicate paste for electrical insulation and this core was packed in an 18" diameter aluminum tube with bubbled alumina insulation. The furnace was horizontally mounted. It can support a 600°C temperature gradient over a 30" distance. Figure 3 shows a schematic diagram.

The temperatures were controlled (± 5°C) with a Honeywell proportional controller using a Type K (chromel-alumel) thermocouple placed on the windings in the hottest zone. The power supply operated at 220 V.

The ends of the furnace were plugged with alumina wool during the heat treatments to prevent large temperature fluctuations resulting from the chimney effect common in open-bore air furnaces.

Other furnaces used in the heat treatments and sample preparation include: a Brew vacuum furnace with a tungsten mesh element and tantalum heat shields; a vacuum furnace with a tantalum strip filament and tantalum heat shields,
and; a vertically mounted Marshall resistance furnace which, as the gradient furnace, has the core open to air. Both vacuum furnaces have a pressure of $10^{-6}$ torr. The tantalum heat shields served as getters for the gaseous impurities and further protected the sample from contaminants. The samples in the open core furnaces were protected by quartz encapsulation for which the details will be given below.

The sample temperatures were monitored with a calibrated Doric 412A Trendicator which can accommodate five thermocouples. Type S (Pt-Pt10%Rh) thermocouples were used for all the samples. The thermocouples were prepared according to NBS specifications and were not calibrated further. An uncertainty in the temperature of ± 5°C is assigned to this technique of thermocouple preparation.

All X-rays were obtained on a Picker model 3488K diffractometer using iron $K_\alpha$ radiation.
Sample Preparation

All of the alloy samples were prepared by standard powder metallurgical techniques. The appropriate amounts of metal powders were mixed in glass jars with ceramic mixing balls on a machine which rotated the jars end over end for at least 6 hrs. The powders were cold-pressed in a 1/2" steel die at 100 Kpsi and the resulting compacts were sintered at 1200°C for 4 hrs in a Brew vacuum furnace which had been evacuated to \(10^{-7}\) torr and then backfilled with argon when at temperature. The specimens were further annealed at 1130°C for 6 days in a tantalum filament vacuum furnace at a pressure of \(<10^{-6}\) torr.

The specimens were analyzed laterally and longitudinally for homogeneity with an AMR scanning electron microscope/energy dispersive X-ray analyzer. The ratio of peak heights for the Co and Mo signals were well within the statistical fluctuations of the instrument. The ratios did not vary systemati
cally as the probe progressed along the sample and the speci
mens were considered to be homogeneous.

The rods were then cut into pellets approximately 1/4" thick. The pellets that underwent heat treatments in the open-bore furnaces were encapsulated in quartz for protec
tion. Each tube was evacuated to \(10^{-3}\) torr and flushed with argon. This procedure was repeated four times. Prior to the final sealing, the argon pressure in the capsule was slightly less than 1 atm. For all experiments except the
initial one in the gradient furnace, a piece of tantalum foil was included in the capsule to act as an internal getter.

The lead used as a catalyst was deoxidized by melting it in a tantalum crucible sealed in a quartz capsule as described above. The two metals are immiscible and tantalum has a much higher affinity for oxygen than lead. The oxygen fugacity in the lead is decreased to approximately $10^{-30}$ atm at 805°C.

The thermocouples were tied to the quartz capsules using either kanthal, chromel, or alumel wire. An indentation in the quartz prevented the sample from moving away from the thermocouple tip during placement in the horizontally mounted furnace. A calibration run showed that this method of positioning the thermocouple measures the sample temperature within the inherent uncertainty of the thermocouples as prepared.

The samples heated in vacuum had the thermocouple tip positioned directly at the sample though separated from it by the 0.04" thick alumina crucible.

The molybdenum powder was -100 mesh, 99.98% pure and the cobalt powder was -325 mesh, 99.999% pure. Both were purchased from Apache Chemicals. The lead was 99.9999% pure, the tantalum foil was 99.9% pure and both were obtained from Lawrence Berkeley Laboratory Stores.

After the sample handling and experiments, the samples were analyzed for typical impurities by atomic absorption spectroscopy in the U.C. Berkeley Chemistry Department Micro-
analytical Lab. Typical impurity levels were 0.08 at% Fe, 0.5 at% Al and 0.03 at% Si.
Gradient Furnace Experiments to Determine the Maximum Transformation Temperature for HCP to FCC Cobalt Solid Solution in the Cobalt-Molybdenum System

The initial experiment was conducted with a15 at% Mo - 85 at% Co alloy. From the suggested phase diagram shown in Figure 2, it is seen that this composition below 1000°C corresponds to a mixture of the \( \kappa \)-Co₃Mo intermetallic compound with either the \( \alpha \)-Co or \( \beta \)-Co solid solution.

Five specimens of this composition were prepared and sealed in quartz capsules as described elsewhere in this paper. The samples were maintained at the following temperatures for 1010 hrs: 555°C, 628°C, 711°C, 843°C and 918°C, and then air-quenched.

Each two-phase sample was X-rayed before and after heat treatment and the differences were noted. The phase behavior was established by comparing the samples' diffraction patterns with those of a \( \kappa \)-Co₃Mo single phase specimen and of the \( \alpha \)-Co and \( \beta \)-Co phases. An etch used by Quinn and Hume-Rothery (17) also proved useful in showing the presence of two phases. It consists of 4 parts of hydrochloric acid, 1 part each of nitric acid, acetic acid and water. After 10 sec, the \( \kappa \)-Co₃Mo phase turns brown, and the grain structure of the solid solution phases appears.

Further heat treatments at 855°C and 875°C showed more precisely the maximum \( \alpha \)-Co to \( \beta \)-Co transformation temperature. These were conducted in a tantalum filament vacuum furnace. Equilibration times were of the order of two and a
half weeks. In order to check for reproducibility, the original as well as additional specimens were used in these subsequent experiments. In all cases the samples were chosen so that a solid-solution phase had to transform, i.e. two samples initially having different solid solution phases in equilibrium with the intermetallic compound were heated until both samples showed identical phases. This demonstrated that solid state equilibrium had been attained.
Experiments with Catalytic Flux to Determine the Composition Range of the HCP Cobalt Solid Solution in the Cobalt-Molybdenum System

The extent of the hcp \( \alpha \)-Co solid solution was investigated using lead to catalyze the sluggish solid state reaction. Cobalt-molybdenum alloys containing 2 at%, 5 at%, 8 at% and 10 at% molybdenum were prepared as before and X-rayed. Prior to encapsulation, however, a small well was drilled in each annealed specimen and approximately 2 g of 99.9999% pure deoxidized lead was placed in the well. See Figure 4. After the encapsulation procedure the samples were placed in the Marshall resistance furnace and maintained at 805°C (± 5°C).

After a week at temperature, the samples were air-quenched and X-ray analysis showed that solid state transformations had taken place.
III. RESULTS AND DISCUSSION

The $\kappa$-Co$_3$Mo intermetallic compound has a primitive hexagonal structure (hP8) exhibiting many X-ray diffraction lines (9,24). The major peaks overlap all of the $\alpha$-Co (hcp, hP2) reflections and many of those of the $\beta$-Co (fcc, cF4) phase. The (200) reflection of the $\beta$-Co phase corresponding to $d = 1.771 \text{Å}$ in pure Co is not overlapped, however, and it unambiguously indicates the presence or absence of that phase.

The initial experiment in the thermal gradient furnace with the 85 at% Co - 15 at% Mo alloy showed that the samples maintained at temperatures of 843°C and below exhibited the hexagonal $\alpha$-Co phase and the $\kappa$-Co$_3$Mo intermetallic compound upon quenching. The sample quenched from 918°C showed the cubic $\beta$-Co phase and the $\kappa$-Co$_3$Mo intermetallic phase. The subsequent heat treatments narrowed the uncertainty in the temperature between 918°C and 843°C and the final heat treatments assign a value for the maximum transformation temperature $T_{tr} = 849°C \pm 15°C$. See Table 2.

X-ray analysis of the four alloys held at 805°C containing 2 at%, 5 at%, 8 at% and 10 at% Mo showed the $\alpha$-Co phase to be in equilibrium with the intermetallic compound in the 5-10 at% molybdenum alloys. The equilibrium phases of the remaining 2 at% Mo alloys are the $\alpha$-Co and $\beta$-Co solid solutions as might be expected from Figure 2.

Two things were accomplished in the experiments:
(1) The stabilization by molybdenum of the low temperature hcp $\alpha$-Co phase, as predicted by the Brewer-Engel correlation, was confirmed. The transformation temperature increases from 430°C in pure Co to a maximum of 849°C in the alloy.

(2) It was shown that the $\alpha$-Co solid solution extends in the phase diagram to between 95 at% and 98 at% Co at 50°C below the transformation temperature of 849°C. At the transformation temperature, the phase field probably extends to no less than 95 at% Co.

The portion of the Mo-Co phase diagram elucidated by this study is shown in Figure 5.

The discrepancy between the earlier results can be reconciled in view of the difficulty in nucleating one solid solution phase in the other phase. This is especially a problem when very pure metals are used in an alloy. A nucleation site is needed and the use of Pb in these experiments provided one. Lead dissolves a very small amount of Mo and 0.48 at% Co at 727°C (25). The slight solubility of Co in Pb permits the Co to adopt its equilibrium structure at the liquid Pb-solid alloy interface and to nucleate the equilibrium phase in the solid. Care must also be taken to prevent oxygen from dissolving in the alloy because it stabilizes the cubic $\beta$-Co phase relative to the hexagonal $\alpha$-Co phase as predicted by the Brewer-Engel correlation. The $\beta$-Co phase has a higher $s,p$ electronic concentration than does
the α-phase and so oxygen, having only s and p electrons, favors the cubic phase. This is the reason why the Ta getters were included in the quartz capsules.
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Figure 2: Co-Mo Phase Diagram given by Brewer et al.
Figure 3: Cut Away View of the Gradient Furnace Windings.
LIQUID Pb

Mo-Co

ALLOY BUTTON

CROSS SECTION OF ALLOY SPECIMEN SHOWING THE WELL FOR THE LIQUID LEAD CATALYST
XBL 817-10470

Figure 4: Schematic of an Alloy Sample with a Well and Liquid Pb.
Figure 5: Portion of the Co-Mo Phase Diagram Elucidated by this Study.
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2. Temperature and Corresponding Equilibrium Phases for 
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### TABLE 1: LOW TEMPERATURE SOLUBILITY PARAMETERS OF SOLID METALS

$$(\Delta E/V)^{1/2} \text{ in (cal/cc)}^{1/2}$$

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TABLE 2: TEMPERATURES AND CORRESPONDING EQUILIBRIUM PHASES
FOR 85 at% Co - 15 at% Mo ALLOY

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REFERENCES

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