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THE SODIUM CHLORIDE - SILVER CHLORIDE
ALLOY SYSTEM

Fourteenth Technical Report

By

R. J. Stokes
C. H. Li

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ABSTRACT

It has been shown that a miscibility gap exists in solid solutions between sodium chloride and silver chloride. At room temperature the two phase region extends from 10% NaCl to 80% NaCl. The precipitation reaction leads to a change in the appearance of single crystals from complete transparency to a milky white opaqueness. Measurements of room temperature mechanical properties have shown a tremendous (100 fold) increase in strength within the two phase region with a slight increase in the rate of work hardening. The transition in slip mode from the coarse wavy slip of silver chloride to the fine straight slip of sodium chloride is found to occur gradually across the diagram.

1. INTRODUCTION

The sodium chloride-silver chloride alloy system is a particularly interesting one to study because of the contrast in physical properties between the two end components. Fundamental differences in their physical properties are discussed in detail in the book by Mott and Gurney[1]. They stem from a number of factors. First, cation diffusion is much easier in silver chloride than in sodium chloride. Second, the Frenkel defect is the predominant type of lattice disorder in silver chloride whereas in sodium chloride the Schottky defect predominates. Third, there is a fundamental difference in their electron band structure due to the higher polarizability and ionization potential of the silver ion. Silver chloride absorbs light at the extreme blue end of the visible spectrum and when irradiated in this range becomes photoconducting. Sodium chloride on the other hand is completely transparent and even when irradiated in the ultraviolet where it begins to absorb does not become photoconducting. The difference in the absorption wavelength cut off and photoconductivity is interpreted in terms of the smaller energy gap between the valence band and the so-called 'exciton' acceptor levels in silver chloride. Thermal excitation of electrons from the valence band into the exciton levels is considered to account for the deep yellow coloration of silver chloride as it approaches the melting temperature[2]. There is no such color change in sodium chloride.

This contrast between sodium chloride and silver chloride also extends to their mechanical behavior. Sodium chloride is extremely brittle and notch sensitive and is prone to cleavage at temperatures almost up to its melting temperature (800 °C), whereas silver chloride under the same loading conditions does not become notch sensitive until the temperature is lowered to room temperature[3]. There is furthermore a distinction between the appearance of slip lines developed on these two solids at room temperature. In sodium chloride the slip lines are straight and very fine; in silver chloride the bands are wavy and coarse with a high surface tilt.

Our interest in the alloy system arose initially from a desire to study its mechanical properties, principally to establish how and when the respective transitions in slip mode and notch sensitivity occurred. At the outset this appeared feasible since both sodium chloride and silver chloride had the same crystal structure with approximately the same lattice parameter (AgC1, $a_0 =$ 5.545Å: NaC1, $a_0 = 5.627$Å) and were reported to form a single solid solution[4, 5, 6]. However, it soon became apparent that this simple phase diagram was inadequate and that there was a solid state precipitation reaction occurring. It is the purpose of this paper to present a fairly detailed analysis of the phase diagram and to describe the precipitation reaction and some of its consequences particularly on the mechanical strength of alloy single crystals.

2. SPECIMEN PREPARATION

The chief difficulty to overcome in the fabrication of dense AgCl-NaCl
alloys was the marked shrinkage on solidification and the general brittle-
ness of the solids involved. The technique which eventually proved most
successful yielded completely dense and fairly homogeneous alloy single
crystals over the whole composition range. First, a liquid melt was pre-
pared in which pure sodium chloride and silver chloride powder (AR quality)
of the appropriate molecular proportions (all alloys are quoted as mole percent
NaCl) were fused together in a Vycor tube. The melt was chlorinated to reduce
any oxides which, if present, wet the Vycor tube causing both the tube and the
ingot to snatter on cooling. Bubbling chlorine gas through the melt also served
to stir it and to maintain a slight excess of chlorine. The melt was poured into
a second Vycor tube so that solidification occurred instantaneously. The cast
ingot at this stage contained a deep pipe, many voids and cracks and was com-
pletely unsuitable for optical examination. Instead it was ground to a powder
fine enough to pass through the narrow constriction of another Vycor tube which
had a sharply pointed tip. The constriction facilitated sealing under vacuum.
The evacuated tube and the powder were then lowered slowly (2 mm hr) through
a furnace having a steep thermal gradient at the melting temperature as in the
conventional Stockbarger technique[1]. Chlorine was evolved on remelting to
maintain a reducing environment in the sealed tube. Ingots produced in this
way were completely dense and within 10% of the end compositions solidified
as transparent single crystals. For intermediate compositions they were not
necessarily single crystals and generally had a pronounced substructure. They
were opaque on withdrawal from the furnace with a milky white appearance. The
reason for the milkiness will be described in the next section. The ingots were
not analyzed chemically after preparation, they were assumed to possess the pre-
determined composition.

Small single crystals suitable for mechanical testing and microscopic examin-
ation were extracted from the ingots by cleavage over {100} planes. Ingots

containing less than 5% NaCl had to be cleaved at liquid nitrogen temperature while higher compositions could be cleaved at room temperature. The opaque crystals were rock hard and extremely brittle which made their cleavage to a perfect orthogonal parallelipiped very difficult. The usual final dimensions were .05" x .05" x .125" although some specimens were larger when the scale of the substructure permitted. They were loaded in compression in an Instron machine at a cross-head deflection rate of .002" min. Because of non-parallel faces many specimens crushed at the corners under load before plastic yielding occurred, this gave a jerky stress-strain relationship. Only those specimens which showed a smooth stress-strain curve were included in the present mechanical results.

Slip band studies were made on both the as-cleaved and chemically polished surfaces. A satisfactory polish was not developed for all alloy compositions. Only the silver chloride rich alloys could be polished reproducibly. They were immersed for three minutes in a warm (50 °C) solution of ammonium hydroxide, rinsed in water and ether and then dried quickly in a hot air stream to avoid any stain. The same technique could not be used for higher compositions since the crystals were slightly soluble in water and the silver and chlorine ions immediately recombined to precipitate silver chloride on the polished surface.

3. THE NaCl-AgCl PHASE DIAGRAM

The phase diagram for sodium chloride-silver chloride alloys determined
in the present work is reproduced in Figure 1. The liquidus and solidus
lines have been constructed from data published by Zemczuzny in 1926[5] to-
gether with several check points, indicated by open circles, taken to confirm
that there was no depression of the melting point. It was found that while com-
plete solid solubility existed across the diagram at temperatures above 170°C,
there was a miscibility gap below this temperature. This type of phase diagram
occurs with gold-nickel and gold-platinum alloys, and for sodium chloride-
potassium chloride[8], sodium chloride-lithium chloride[9] alloys. A miscibility
gap is normally associated with a depression of both the liquidus and solidus lines
to a congruent melting point. In the present system this was not observed, in-
stead the lines were distorted from the usual 'cigar' shape and the solidus line
in particular was flat up to 45% NaCl.

The existence of a two phase region accounts for conflicting observations reported
in the literature concerning the structure of sodium chloride-silver chloride solid
solutions. One source[6] describes x-ray measurements of a single solid solution
for which the lattice parameter changes linearly according to Vegards law.
Another[10] mentions the coexistence of two phases in annealed crystals each
slightly displaced from pure sodium chloride and silver chloride. It will be
shown later that both patterns may be produced by different heat treatments as
may be anticipated from the phase diagram.

The solid solubility boundary in Figure 1 was determined as follows. A small
single crystal specimen of each composition represented by points in the figure,
was placed in an air oven and the temperature varied above and below the solu-
bility boundary. Above the boundary the crystals were completely transparent
and remained so for prolonged periods whereas below the boundary they became
opaque with a milky white appearance. Figure 2 compares two companion single
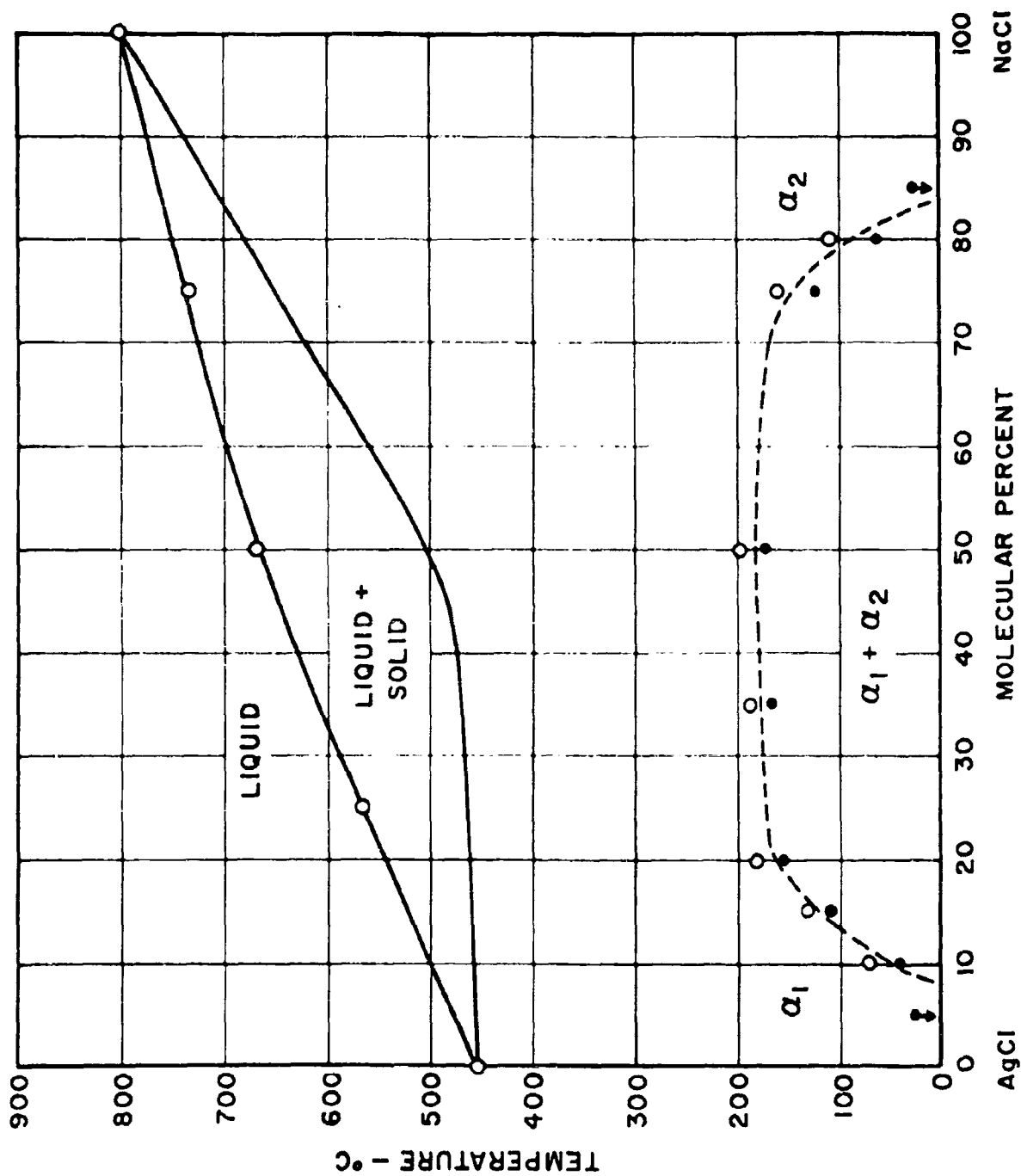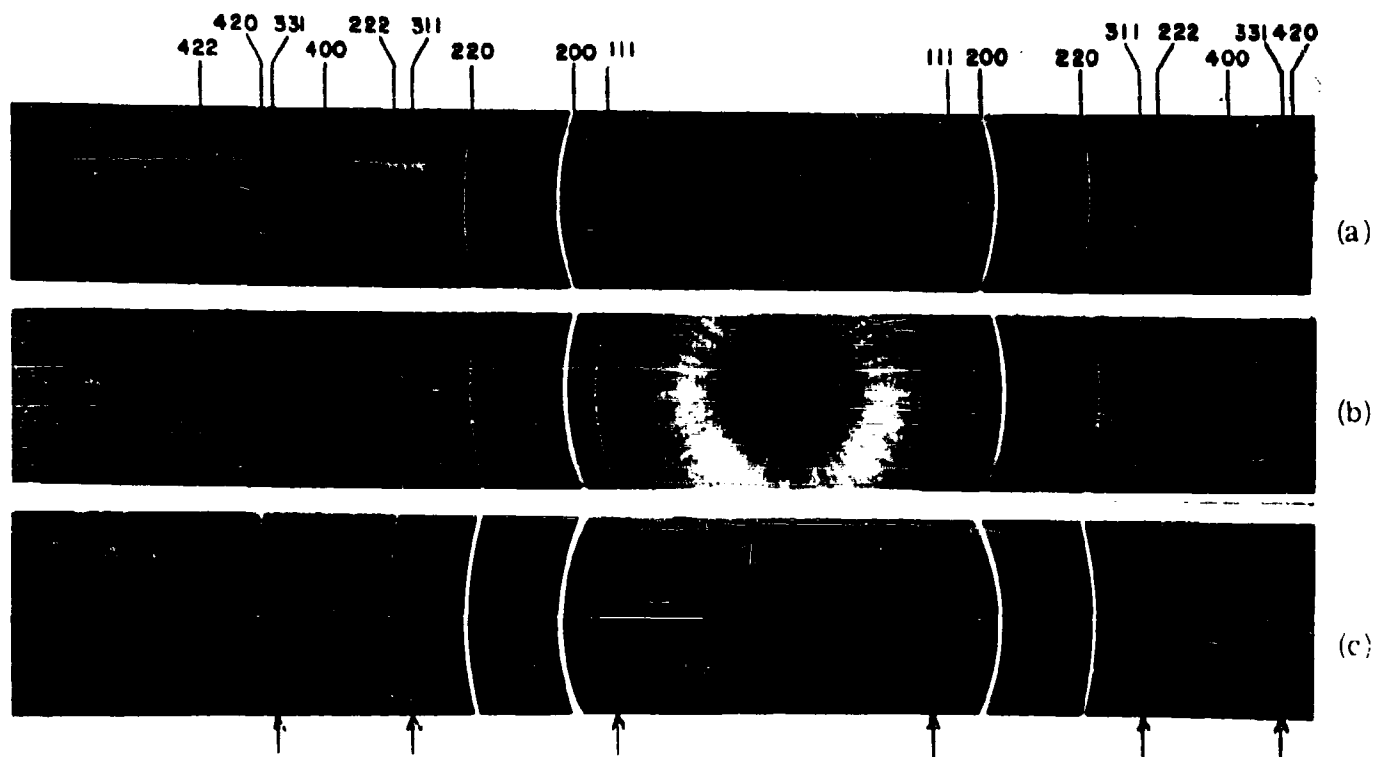
Figure 1 - THE AgCl-NaCl PHASE DIAGRAM

Figure 3 - X-RAY POWDER PATTERNS OBTAINED ON NaCl - AgCl ALLOYS.
(a) 50% NaCl - SOLUTION HEAT TREATED, (b) 50% NaCl - FULLY
PRECIPITATED, (c) 85% NaCl - SOLUTION HEAT TREATED.
ARROWS INDICATE MISSING LINES.

was obtained from a crystal quenched from 200 °C and the lower pattern (Figure 3(b)) from a crystal annealed at 100 °C for 48 hours to establish equilibrium. As expected, Figure 3(a) showed single lines corresponding to a rock salt structure with lattice parameter a_o - 5.584Å, almost midway between the two pure solids. This was in agreement with Vegards law and the measurements of LeBlanc and Quenstadt[6].

The lines in the lower pattern (Figure 3(b)) on the other hand were nearly all doublets in agreement with the observations of Broome[10]. One set of lines corresponded to a lattice parameter a_o 5.540Å, the other to a_o = 5.602Å. According to the phase diagram determined by optical means in Figure 1, the two phases in equilibrium at this temperature should be 15% and 80% NaCl alloys and assuming Vegards law to hold the lattice parameters should be 5.555Å and 5.610Å respectively. It was considered that the agreement was satisfactory.

One feature of the phase diagram was the flatness of the two solid phase equilibrium boundary between 20% and 70% NaCl. To confirm this a solution heat-treated 50 % NaCl specimen was annealed just below its solubility temperature at 165 °C. The Debye-Scherrer pattern again showed doublets corresponding to 20% and 80 % NaCl structures, both well on either side of 50% NaCl in agreement with the phase diagram.

The fact that certain lines in Figure 3(b) were single and not double was most interesting. It was found that all the odd index lines were single and the even index lines double. This occurred because the odd index lines were completely absent from the pattern corresponding to the 80% NaCl structure. This may be explained in terms of the structure-factor formula for the rock salt lattice. It can be shown that the intensity (I) of odd index lines in sodium chloride for example is proportional to:

$$I \propto (f_{Cl} - f_{Na})^2$$

where f_{Cl}, f_{Na} are the atomic scattering factors for chlorine and sodium ions respectively. If now it is assumed that sodium chloride-silver chloride solid solutions consist of silver and sodium ions arranged randomly over the <u>cation</u> lattice sites only, then the intensity is proportional to:

$$I \propto \left[f_{Cl} - \left(\frac{K f_{Na} + (100 - K) f_{Ag}}{100} \right) \right]^2$$

where K is the molecular percent NaCl in solid solution. Since f is proportional to the atomic number then it can be shown that I becomes zero when K $\approx$ 83.3% NaCl. Essentially at this composition the effective scattering powers of the chlorine anion lattice and the randomly mixed cation lattice are equal and the structure appears simple cubic. To confirm this interpretation of the line structure in Figure 3(b), powder patterns were compared for a number of solution heat treated and quenched alloys across the phase diagram and it was shown that the intensity of the odd index lines did indeed reach a minimum at 85% NaCl. The powder pattern for a quenched 85% NaCl alloy is reproduced in Figure 3 (c). A similar effect occurs for the powder pattern of potassium chloride where the atomic scattering factors of the potassium and chlorine ions are again almost equal[11]. The occurrence of the minimum at the predicted 85% NaCl composition strongly indicated that in solid solutions the sodium and silver ions were arranged randomly over the cation lattice sites with no strong tendency for ordering to occur.
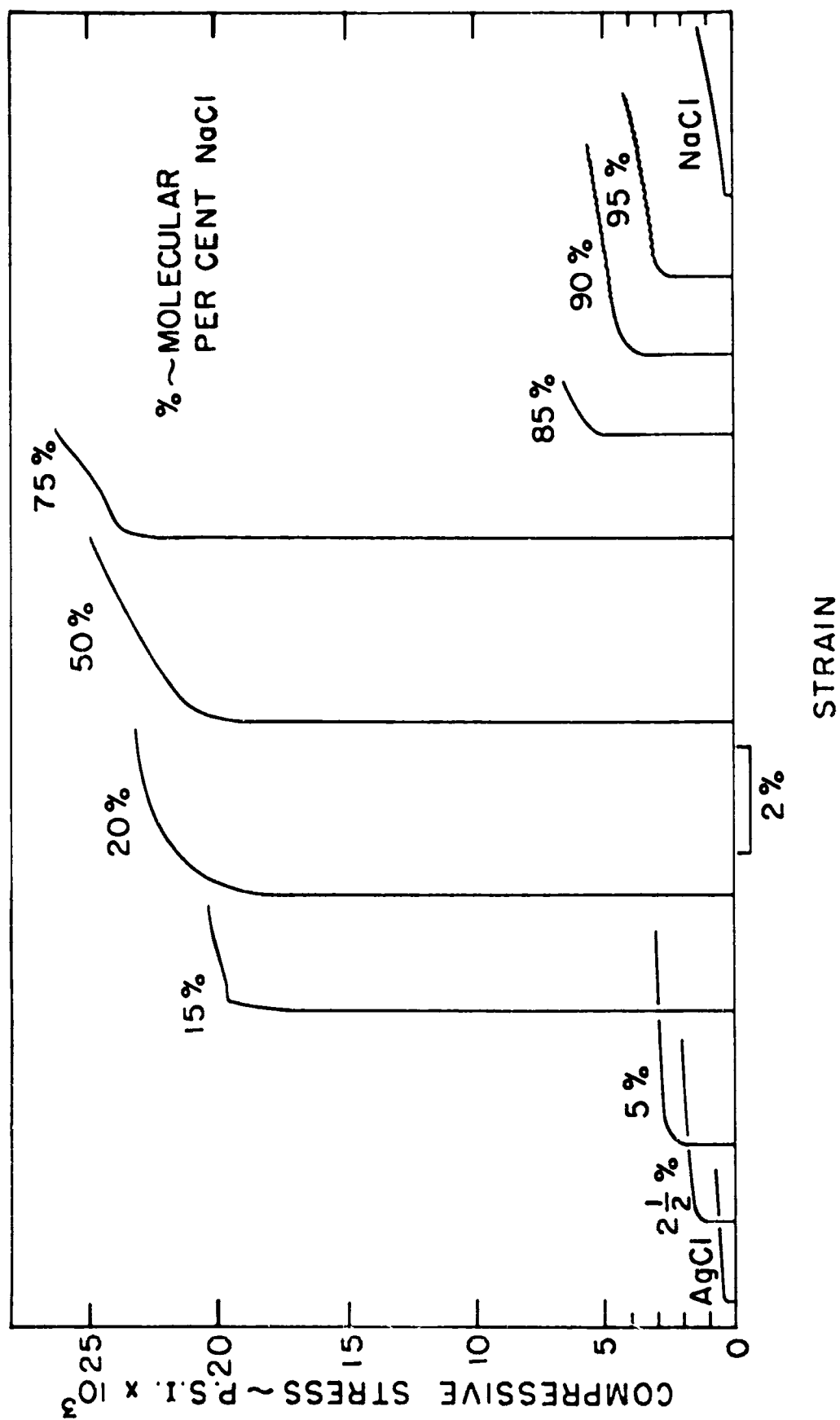
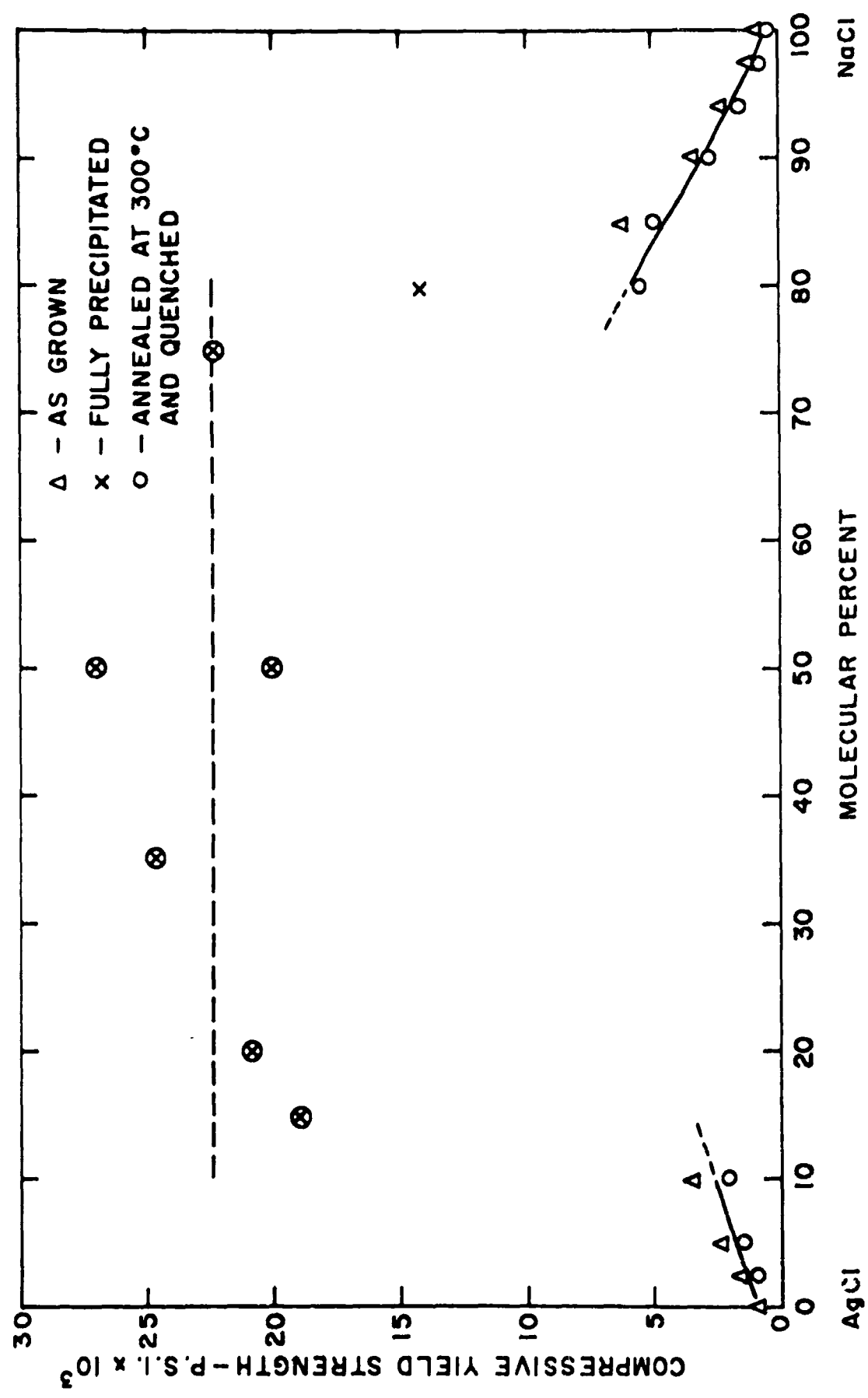Figure 4 - COMPRESSION STRESS-STRAIN CURVES FOR NaCl-AgCl ALLOY SINGLE CRYSTALS

Figure 5 - COMPRESSIVE YIELD STRENGTH VS COMPOSITION

For the 80% NaCl alloy, the strength depended upon heat treatment. Crystals were tested in both the fully precipitated and in the solution heat treated condition. Solution heat treatment consisted of 1/2 hour anneal at 300 °C followed by an air quench, the precipitation treatment consisted of an anneal for a much longer period (approximately 1 week) at 40 °C. As Figure 5 shows, the fully precipitated 80% NaCl alloy had a strength of approximately 15,000 psi compared to 5,000 psi when solution heat treated. However, when similar heat treatments were applied to other crystals of the intermediate composition range there was no detectable lowering of the yield strength after solution heat treatment. In addition, there was no indication of softening due to overaging following prolonged storage at room temperature as might be expected if the tremendous increase in strength was due to precipitation hardening alone.

The nature of the strengthening mechanism for the intermediate compositions is the subject of a more detailed investigation of the mechanical properties of these alloys. The slight difference in lattice parameter between the two phases in equilibrium and the fact that they have the same crystal structure would favor the formation of a coherent precipitate, which is well known to be the most effective source of precipitation strengthening in metal alloy systems. However, there is still the need to explain the equally high strength of solution heat treated alloys. As described in Section 3, they take many hours to manifest precipitation at room temperature and would be expected to be much softer than the fully precipitated material, at least in the very early stages.

It is considered that the linear increase in strength for dilute alloys is caused by solid solution strengthening as has been observed for other single phase silver chloride alloys[12].

6. SLIP BAND STUDIES

Most solids of the rock salt structure, including sodium chloride, lithium fluoride and magnesium oxide, show slip bands at room temperature which are extremely straight and fine (see for example Figure 6(c)). The fineness may be understood as follows. Individual bands in these solids become filled with dislocations and further deformation progresses by a widening of the bands due to dislocation movement and multiplication at their edge only. The shear strain within the bands at saturation is generally quite low and there is a correspondingly small tilt produced on the specimen surface. The edges of the slip bands appear therefore as very fine lines. In silver chloride, by contrast, the shear strain within the band is far greater and the slip bands appear much coarser due to the high surface tilt (see for example Figure 6(a)). In addition they are macroscopically wavy when viewed normal to the $\langle 110 \rangle$ slip direction.

The appearance of slip bands on solution heat treated alloy crystals was determined across the whole composition range between sodium chloride and silver chloride to ascertain the manner of transition from one characteristic slip mode to the other. All alloys maintained the $\langle 110 \rangle$ slip direction and there was a fairly gradual transition in the slip band appearance. The coarse wavy slip illustrated in Figure 6(a) for a 2.5% NaCl alloy persisted up to 10% NaCl. However there was a decrease in the amount of forking and by 15% NaCl the slip bands became noticeably straighter and finer. Slip bands developed on a 35% NaCl alloy are illustrated in Figure 6(b). They were virtually straight although cross slip could still be observed. These slip bands differed from those of Figures 6(a) and 6(c) in that they were not continuous across the specimen but often occurred as short ripples in the surface. At 50% NaCl the slip bands were slightly wavy as in Figure 6(b) but the surface tilt was less. Slip bands produced on the 75% NaCl alloy in Figure 6(c) were absolutely straight and very fine exactly similar to those produced on pure sodium chloride at room temperature.

Slip bands were difficult to resolve on the fully precipitated crystals presumably because of the scale of the microstructure.

7. SUMMARY

It has been shown that a miscibility gap exists in solid solutions between sodium chloride and silver chloride. At room temperature the gap extends from 10% NaCl to 80% NaCl. The precipitation reaction within the two phase region results in a change in the appearance of single crystals from complete transparency to a milky white opaqueness, the rate of change depending upon the temperature. At room temperature the change takes place over a period of two to three weeks. Measurements of the room temperature compressive yield strength of alloy single crystals have indicated a sudden and spectacular increase in strength within the two phase region with a slight increase in the rate of work hardening. This strengthening occurs independently of the heat treatment applied to the crystals. The transition in slip mode from the coarse wavy slip of silver chloride to the fine straight slip of sodium chloride occurs gradually across the diagram for solution heat treated crystals.

When sodium chloride is alloyed with other alkali chlorides it shows a consistent tendency to form a miscibility gap. With lithium and potassium cations the ionic size difference ($\sim 25\%$) is probably the chief factor determining segregation. On the other hand sodium and silver have approximately the same ionic size (within 2%) and the differences in their physical nature must be the determining factor.

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