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DEVELOPMENT OF A-15 (V_3Ga) SUPERCONDUCTING MATERIAL
THROUGH CONTROLLED PRECIPITATION

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ABSTRACT

A monolithic process has been used to produce superconducting materials by controlled precipitation of A-15 (V_3Ga) phase inside the B.C.C. matrix of a V-rich solid solution. Ingots of V-17~19 at. % Ga were prepared by arc-melting, homogenization, and quenching, were deformed at intermediate temperature into tape, and were aged at temperatures in the range 600-1000°C to precipitate the A-15 phase. The maximum critical transition temperature ($\sim 14.8^\circ K$) was found in materials aged at temperatures of 750°C or below. At these aging temperatures the T_c initially increases with aging time, and passes through a distinct maximum. The source of the exceptionally high T_c is not clear, but may be attributable to a combination of high solute content and internal strain in the small A-15 precipitate particles.

INTRODUCTION

Intermetallic compounds of the A_3B type which form in the A-15 crystal structure often have excellent superconducting properties. However, these compounds are intrinsically brittle, and special processing techniques must be used to form them into superconducting wires or tapes. A number of manufacturing techniques have been suggested for laboratory or commercial use. These include the well-developed "bronze" process, which is widely used in the manufacture of Nb_3Sn ^{1,2} or V_3Ga wires^{3,4}, "in situ" multifilamentary wire processing⁵⁻⁸, infiltration method⁹, chemical vapor deposition¹⁰, powder metallurgical (P/M) processing^{11,12}, and others. While considerable success has been obtained with these various manufacturing methods, they have the common drawback of being metallurgically elaborate and difficult to accomplish in practice.

The metallurgically simplest method of wire manufacture involves casting an ingot of the desired composition and extruding or drawing the cast material into a wire. Virtually all conducting wires, including ductile Nb-Ti superconducting wires, are manufactured in this way. It is possible that variations on conventional wire making practice may also be used for the manufacture of high T_c superconducting wires or tapes^{13,14}.

A monolithic processing technique which should succeed in producing superconducting wires containing A_3B phases is simple to design in theory, and is illustrated in Fig. 1 with reference to the V-Ga phase diagram^{15,16}. The V_3Ga phase is an ordered phase which is present in the equilibrium phase diagram in the isolated region shown to the left hand side. The region of preference of the V_3Ga phase lies below a well-defined critical temperature, above which V and Ga form a solid solution. One may hence design a monolithic process in which a V/Ga ingot of composition near

V_3Ga is made homogeneous by annealing at temperatures above the ordering temperature. If the ingot is subsequently cooled at a rate sufficient to suppress the formation of the V_3Ga ordered compound, a supersaturated solid solution of Ga in V will be obtained. The solution should be relatively ductile and amenable to processing through wire drawing or extrusion. The superconducting phase might then be introduced by heat treatment at a temperature below the ordering temperature but high enough for the precipitation of the V/Ga phase to proceed at a kinetically reasonable rate.

There is, moreover, good reason to suspect that the mechanical deformation involved in the wire drawing, extrusion or tape rolling step would have a favorable influence on the ultimate properties of the material. The A-15 V_3Ga structure, which has a rather large mismatch with the parent V/Ga lattice, should form preferentially along dislocation lines within the deformed matrix phase, promoting a closely interconnected structure of the superconducting elements. A process of this type is most straightforward in the V-Ga system, whose phase diagram contains a solid solution region above the ordered V_3Ga compound, but modifications of it could easily be designed for other systems of the A_3B type.

While the monolithic processing procedure described above is simple and appealing in theory, and has been used with some success in exploratory research by Pan, et al.¹³, its practical implementation faces substantial difficulty. The three most obvious problems are the following, which will again be discussed in terms of the V/Ga system.

(1) Quench-cracking. Assuming that one can achieve a homogeneous solid solution of Ga and V by annealing above the ordering temperature, one must then achieve a homogeneous solid solution at room temperature,

which requires that the ingot be cooled sufficiently rapidly to suppress the formation of the A-15 phase. However, the rapid cooling of alloys frequently results in a phenomenon known as quench-cracking, in which the alloy spontaneously fractures due to thermal stresses on cooling. This problem is usually attributed to chemical segregation during high temperature annealing, and special steps must sometimes be taken to overcome it. Quench-cracking-like phenomena have, in fact, been encountered in previous attempts to form A_3B superconducting compounds by monolithic processes¹⁷.

(2) Low Temperature Brittleness. Assuming that the sample has been successfully quenched to form a homogeneous solid solution at low temperature, it still does not follow that the sample can be successfully formed into a wire or tape. The matrix phases of interest, V or Nb, are bcc-structure refractory metals which typically have high ductile-to-brittle transition temperatures, with the result that their solid solutions may prove to be extremely brittle at low temperature.

(3) Non-stoichiometric Precipitation. The A_3B compounds of interest as high field superconducting phases permit relatively large deviations from stoichiometry, as indicated by the breadth of the stability field of the V_3Ga in the V-Ga phase diagram. It is well-known that the superconducting properties of the A-15 compounds deteriorate as the compounds deviate from the true A_3B composition. It may, therefore, prove difficult to achieve good superconducting transition temperatures if the alloy is intentionally made lean in solute so as to facilitate melting and wire and tape fabrication.

It is the purpose of this paper to describe how these three problems may be overcome, at least in the laboratory sense, to achieve V/Ga wires

and tapes having very promising superconducting transition temperatures.

EXPERIMENTAL PROCEDURE AND RESULTS

Melting, Homogenization and Quenching Procedure

The alloys investigated here were prepared by arc melting pure starting components under an argon atmosphere. The starting materials were V, 99.8% pure, purchased from the United Mineral and Chemical Corporation, Ga, 99.999% pure, purchased from the Orion Chemical Company. The arc furnace employed a water-cooled non-consumable tungsten electrode and a copper hearth. The atmosphere was high purity argon, 99.995%, which was passed over heated zirconium to remove residual oxygen and nitrogen, and through a cold trap to condense moisture before being introduced into the furnace. During melting, the samples were inverted on the copper hearth and remelted at least four times to achieve complete alloying between the V and Ga. There is, of course, a loss of Ga during melting through vaporization. The composition of the starting mixture of V and Ga is chosen so as to achieve a desired Ga content after melting and homogenization. The nominal composition of the final product is then calculated on the assumption that the observed sample weight loss was due to the vaporization of Ga. This method of computing the nominal composition appears reasonable as evidenced by chemical analysis, which yields Ga contents differing by no more than a fraction of an atom percent from those computed.

The samples investigated in this work had nominal compositions in atom percent, V-17~19at% Ga. Referring to the V-Ga phase diagram, Fig. 1, samples of these compositions should preferentially form homogeneous bcc solid solutions at temperatures above approximately 1300°C. The melted ingots were hence homogenized at a temperature of 1350°C for 24 hours to

achieve a homogeneous starting material. During homogenization the samples were wrapped in tantalum foils and encapsulated in quartz tubing. There is a slight further loss of Ga during the homogenization treatment, and this loss is accounted for in computing the final composition.

Following homogenization the samples were quenched in water at room temperature. No quench-cracking was observed during quenching of the samples. Moreover, both optical microscopy and x-ray diffraction indicated that the precipitation of the A-15 phase was suppressed during the quench. The suppression of precipitation was further confirmed by superconducting transition temperature measurements, using the inductive method with a calibrated germanium resistance thermometer, which showed no critical transition temperature (T_c) above 5°K. The homogenization plus water-quenching treatment hence seemed successful in avoiding both quench-cracking and precipitation of the A-15 phase.

Mechanical Deformation

In the homogenized and quenched condition the V/Ga samples were brittle, and fractured catastrophically on deformation at room temperature. A scanning electron fractographic analysis of broken sample surfaces (Fig. 2) revealed that the fracture mode was almost entirely quasi-cleavage, indicating that the brittleness of these samples is due to the normal low temperature brittleness of the bcc matrix structure. The ductile-to-brittle transition temperature of bcc alloys is known to increase with grain size. Optical microscopy revealed that during the homogenization treatment substantial grain growth had occurred, giving a typical sample grain size larger than 1 mm.

To avoid low temperature brittleness, the samples were deformed at ~800°C. The total reduction in thickness accomplished during the warm

rolling was $\sim 75\%$.

Examination of the specimens after warm reduction by 75% at 800°C revealed that there is a slight precipitation of the A-15 phase during mechanical deformation. A measurement of the superconducting transition temperature of the specimens after warm deformation gave $T_c \sim 9-10^\circ\text{K}$.

Precipitation of the A-15 Phase

Following warm deformation the samples were aged to precipitate the A-15 phase. The aging treatments were conducted over a range of temperatures from 600°C to 1000°C and for a number of aging times. The superconducting transition temperatures of the product were then measured as a function of aging temperature and time, again using the inductive method. In the case of samples aged at lower temperatures and shorter times, a broad range of transition temperatures was observed. This transition temperature range becomes narrower with longer aging time.

The onset superconducting transition temperature is plotted as a function of aging temperature and time for the V-18.5 at. % Ga samples in Fig. 3 and for the V-17.5 at. % Ga samples in Fig. 4. For both compositions it proved possible to obtain transition temperatures in excess of 14°K by aging the deformed samples at temperatures between 600°C and 750°C . The maximum T_c measured was $\sim 14.8^\circ\text{K}$, a value which compares favorably to the 15.4°K which is believed to be the maximum attainable critical temperature for the stoichiometric V_3Ga composition¹⁸. For aging temperatures of 800°C or below there is an apparent maximum in the curve of critical temperature versus aging time. The aging time required to reach this maximum increases dramatically as the aging temperature is lowered below 750°C . However, in the case of the 18.5 at. % Ga

specimen the peak critical temperature is reached after only three hours aging at 750°C. With aging temperatures of 800°C to 1000°C the critical temperature decreases to a value of 10°K or below when the aging time is made long.

Optical microscopic examination of these specimens reveals evidence for the precipitation of the A-15 compound in samples aged at 800°C to 1000°C (Fig. 5a). However, samples aged at 750°C or below for the shorter aging time do not show optical evidence of the A-15 precipitate (Fig. 5b) beyond the initial precipitation which could be observed after the deformation step. Transmission electron microscopic analysis of these samples are in progress to identify precipitates at higher magnification.

DISCUSSION

It seems best to discuss these results in reverse order, beginning with the interesting dependence of the measured T_c on the aging temperature and time.

The Superconducting Critical Transition Temperature

Reference to the equilibrium phase diagram shown in Fig. 1 reveals that for the sample compositions used here (17-19 at. % Ga) and for aging temperatures in the range 600°C to 1000°C the specimens fall within a two-phase region. At equilibrium these samples would therefore be expected to contain an A-15 phase, of composition determined by the edge of the two-phase region at the particular aging temperature. At 1000°C the predicted composition of the A-15 phase is ~ 20 at. % Ga, which, according to the work of Das et al.¹⁶, has $T_c < 10^\circ\text{K}$. Because the equilibrium concentration of the A-15 phase becomes progressively leaner in Ga as the aging temperature is decreased, successively lower transition temperatures would be expected from equilibrium considerations. However,

under the non-equilibrium conditions, established during initial aging in the present work, the reverse happens. While aging at 1000°C eventually leads to a T_c near 8°K, and aging at 800°C also eventually yields a rather low T_c , the maximum value of T_c increases when lower aging temperatures are used.

The observation of higher T_c is almost certainly associated with the small size of the initial precipitate particles and with the high degree of supersaturation under which they form. Preliminary transmission electron microscopic analysis of the specimens aged at lower temperatures to near maximum T_c does show diffraction evidence for A-15 precipitates within the V/Ga matrix. However, the precipitates are extremely fine and have not yet been clearly resolved.

The small A-15 particles which initially precipitate from the supersaturated parent matrix will be expected to differ from the equilibrium A-15 phase in two aspects; composition and state of internal strain. Both differences may contribute to the high transition temperatures observed.

There are thermodynamic reasons to anticipate that the severe supersaturation present at the time the particles form may cause them to have a more nearly stoichiometric composition than the equilibrium phase diagram would suggest. The thermodynamic argument follows directly from Gibbs¹⁹ who pointed out that the energetic barrier to the formation of a small particle of a new phase within a supersaturated matrix is minimized when the new phase forms in a state such that the chemical potential of each component remains constant. If both the parent and product phases are incompressible then the relevant chemical potential is the relative chemical potential, which is equal to the slope of the free energy versus

composition curve of the parent solution measured at its mean composition, as illustrated in Fig. 6. The most favorable composition of small precipitate phase is that composition at which its free energy vs. composition curve, at the same temperature, has an equal slope. The predicted result, illustrated in Fig. 6, is that small precipitate particles formed under significant supersaturation will tend to be richer in solute than indicated by the equilibrium phase diagram. If the free energy vs. composition curve of the precipitated phase has a strong minimum near its stoichiometric composition, in the sense that the curvature of the free energy curve is very high, then the precipitate will have a thermodynamic tendency to form initially in an almost stoichiometric composition. This thermodynamically preferred solute enrichment of the initial precipitate particles currently seems to offer the most plausible explanation for their very high transition temperatures.

Since the A-15 phase is in this case formed by the solid state precipitation within the solution matrix, and since the A-15 phase has a substantial mismatch with the parent matrix, one would also anticipate that the initial precipitates would be under a relatively high state of strain. The internal strain of the precipitate particles may be relieved somewhat by preferential nucleation along defects in the matrix such as dislocations or dislocation modes and may be further relieved by the further accumulation of thermal vacancies at the precipitate-matrix interface. It still must be expected that particles at relatively low temperature will retain some strain for rather long aging time. The internal strain is known to influence the critical transition temperature in ways which remain poorly understood²⁰⁻²². According to T. F. Smith²⁰, however, the T_c of A-15 (V_3Ga) increases with hydrostatic pressure. While the effect measured by Smith is small ($\partial T_c / \partial p = 1.05 \times 10^{-5} \text{K/bar}$),

the result does suggest that internal strain may also contribute to the high value of T_c .

On the basis of the above reasoning, the variation of critical temperature with the time and temperature aging may be rationalized as follows. In the earliest stages of aging at all temperatures, small precipitates form which are nearly stoichiometric in composition but are too small size to achieve an optimum critical temperature. As aging proceeds, the small precipitates coarsen, essentially at constant supersaturation, so that the critical temperature increases rapidly. Eventually a growth of the precipitate particles and the relief of the supersaturation of the parent matrix causes the composition of the particles to adjust towards the equilibrium value, with a gradual loss of critical temperature. If the aging temperature is high, 800°C or higher, the "over-aging" adjustment toward an equilibrium composition sets in before a peak critical temperature is reached, so that the maximum critical temperature is rather low. At lower temperatures a critical temperature near the optimum value for the V_3Ga phase is approached and maintained for some time before over-aging gradually drives the system toward equilibrium. This interpretation is, in part, testable through high-resolution electron microscopy. The relevant experiments are tedious, but are now in progress.

Formation and Deformation of V/Ga Solid Solution

To form a wire or tape from V/Ga solid solution, it is necessary to prevent spontaneous cracking of the V/Ga ingot during initial quenching, that is, to suppress quench-cracking, and it is further necessary to identify procedures for rolling or drawing the ingot without cracking after it has been cooled.

In the case of the V/Ga solid solution, there are three potential sources of quench-cracking which must be guarded against. The first is the formation of brittle interdendritic compounds during the solidification, in this case Ga-rich intermetallic phases. The second is the segregation of embrittling species to grain boundaries during homogenization; and the third is the precipitation of A-15 phase along grain boundaries during quenching.

To guard against the presence of crack-inducing interdendritic compounds, the alloys were homogenized within the solid solution field in the phase diagram for an extended period. Given the high purity of the starting materials no grain boundary segregation was anticipated during this homogenization treatment, although it should be mentioned that quench-cracking presumably associated with an oxygen segregation to the grain boundaries has been encountered in other phases of this project in the samples containing unusually high oxygen concentrations after melting. Following homogenization a normal water quench appears sufficient to suppress precipitation of the A-15 phase to any significant extent. With the exception of the oxygen-containing sample mentioned above no quench-cracking was encountered in this research.

In order to draw the supersaturated solid solution into a wire or tape, however, it did prove necessary to use warm working treatments at approximately 800°C. Attempts to deform the samples at room temperature encountered severe brittleness problems. This result was not surprising since the V/Ga matrix is a body-centered cubic phase which is known and expected to undergo classic ductile-brittle transition behavior as the deformation temperature is lowered. The ductile to brittle transition

temperature in body-centered cubic metals is known to be a strong function of the alloy grain size. Due to the extensive homogenization of the samples, the grain size of the alloy in the as-quenched state is large, approximately > 1000 microns. The ductile-brittle transition temperature is therefore high, and warm working procedures are necessary if the alloy is not to crack during deformation. Lower working temperatures could presumably be used if the alloy is treated so as to refine its initial grain size, either by making chemical additions to inhibit grain growth during the homogenization step, or by deforming and recrystallizing the alloy prior to the principal working operation. Research along these lines is currently in progress.

CONCLUSIONS

The principal conclusion of the present research is that it is possible to produce high T_c wires or tapes from the V-Ga system by a monolithic process in which an ingot is cast as a supersaturated solution of V and Ga, homogenized at elevated temperature, quenched to preserve the supersaturation, formed into a wire or tape, and finally heat treated at a relatively low temperature to precipitate the superconducting phase. To achieve exceptionally high critical temperatures, the precipitation reaction must be carried out at temperatures below approximately 750°C. The measured critical temperature then becomes a function of the aging time, and reaches a maximum value as high as 14.8°K in alloys containing 17-19 at. % Ga. The reason for the exceptionally high critical temperature when the precipitation is carried out at lower aging temperatures is not established, but may be plausibly interpreted as due to a thermodynamic tendency for small precipitates formed from highly

supersaturated solid solutions to be rich in solute content. The effect is possibly enhanced by internal strain of the initial precipitate particles.

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FIGURE CAPTIONS

- Figure 1. The relevant portion of the V-Ga equilibrium phase diagram as reported by van Vucht et al.¹⁵ (dot-dash lines) and modified by Das et al.¹⁶ (solid lines).
- Figure 2. Scanning electron fractograph of a V-17.5% Ga sample broken after homogenization and quenching. The fracture occurs primarily through transgranular cleavage.
- Figure 3. The critical transition temperature of deformed 18.5% Ga samples as a function of aging time for various aging temperatures.
- Figure 4. The critical transition temperatures of deformed 17.5% Ga samples as a function of aging time for various aging temperatures.
- Figure 5. Optical micrographs of V-17.5% Ga samples aged for 10 hours at (a) 800°C and (b) 700°C showing the precipitate phase.
- Figure 6. Schematic illustration of Gibbs free energy vs. composition at given temperature. C_1 and c_2 are the equilibrium compositions of the hypothetical α and A-15 phases at this temperature. C_1^* is the composition of the supersaturated solid solution and c_2^* is the preferred composition of the initial A-15 precipitate.

Figure 1.

XBL 793 - 5872

Figure 2.

XBB 798-10315

Figure 3.

XBL798-6765

Figure 4.

XBL 793-5871

Figure 5a

XBB 798-10375

Figure 5b

XBB 798-10374

XBL 793-5877

Figure 6.

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