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The Metallurgy of Remendur: Effects of Processing Variations

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A recent development effort in telecommunications switching apparatus has been directed toward the production of a remanent reed, dry, sealed contact (remreed). Remendur, a medium-hard magnetic alloy nominally composed of equal parts iron and cobalt and 2.7-wt. percent vanadium, was chosen as the reed material in this contact. However, the application required the alloy to possess rather specific magnetic and mechanical properties and considerable difficulty was experienced in consistently processing Remendur into wire with these specified properties. To ascertain the sensitivity of these properties to variations in processing times and temperatures, and vanadium content, two melts of Remendur (2.5-percent V and 3.0-percent V) were processed with selected alterations in annealing temperatures at several stages. Microstructures were characterized following each step by light microscopy and were correlated with the appropriate ternary equilibrium diagram. Results demonstrate that microstructures developed by anneals between 900°C and 950°C are extremely sensitive to the precise temperature of the anneal and composition of the alloy. The microstructure, which strongly influences magnetic and mechanical properties, can be varied over the limits of the two-phase $\alpha_1 + \gamma$ *region by variations in vanadium content of only 0.5 wt. percent and by the small 50°C temperature range.*

I. INTRODUCTION

Historically, considerable difficulty has been experienced in consistently processing the Fe-Co-2.7-wt. percent V alloy (Remendur) into wire with specified magnetic and mechanical properties. A major cause for these problems has been that the metallurgy of this ternary system was not sufficiently understood. A number of investigations have provided information on various aspects of phase equilibria[1–5] and kinetics and mechanisms of phase transformations[6–10] in this system.

The most notable of these are the work of Köster and Schmid[1] on the phase equilibrium of the system and the work of English[6] and Martin and Geisler[2] on the (FCC) $\leftrightarrows$ (BCC) transformation. However, discrepancies still exist on aspects of phase equilibrium,[1,2,4,7] mechanical ductility,[2,6,7,11] transformation kinetics,[2,6,7,10] and the influence of these factors on the development of magnetic properties.[2,6,7,10,12,13] These discrepancies made analysis of the sensitivity of the mechanical and magnetic properties to composition and to heat treating times and temperatures during processing unreliable with further investigation. This analysis is vital since 2.7-percent V Remendur is being used as the reed material in the remreed contact.[14] This paper describes the characterization of low-vanadium (2–3-wt. percent) Remendur, mainly by light microscopy, for the various processing steps from 6.35-mm (0.25-inch) rod through 0.53-mm (0.021-inch) wire to 0.18-mm (0.007-inch) flattened strip. Correlation of the microstructures with the equilibrium phase diagram of Köster and Schmid[1] is provided. Aspects of the microstructures in relation to cold ductility and magnetic properties are discussed.

II. MATERIALS AND EXPERIMENTAL PROCEDURES

Two melts of Remendur, each with a different vanadium content, were used in these experiments. The alloys were prepared by Battelle Memorial Institute with nominal compositions of 2.5-percent V-balance equal Fe/Co and 3-percent V-balance equal Fe/Co. The actual analyses as determined by atomic absorption spectroscopy are given in Table I. The typical Remendur processing sequence from melting through final reed fabrication is outlined in Fig. 1. The influence on cold ductility, yield and tensile strength, resistivity, and magnetic properties of several steps in this processing sequence was unclear. These steps included temperature of and rate of cooling from the 6.35-mm rod anneal, the need for intermediate strand anneals, and the temperature of the 0.53-mm wire strand anneal. Also, the influence of the stamping operation relative to the unworked shank on final reed properties was of interest.

TABLE I—REMENDUR COMPOSITIONAL ANALYSES
(wt. %)

	V	Co	Fe
3% V Battelle	2.97	48.70	47.34
2.5% V Battelle	2.46	48.36	48.27

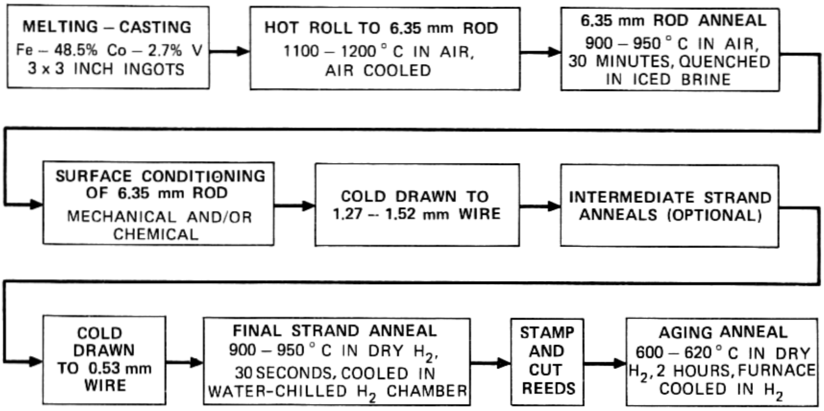

Fig. 1—Typical Remendur processing.

An experimental program was developed to provide clarification of the influences of these factors. A synopsis of this program is given in Fig. 2. It involved carrying two compositions of Remendur through the entire processing sequence with appropriate variations in parameters at each critical stage. For temperature variation, a matrix of temperatures between 900°C and 950°C was investigated. This range was based on previous statements by Gould and Wenny that cold ductility could be obtained by an iced brine quench from 925°C[12] and the current use of this temperature by K. Olsen (Bell Laboratories–Murray Hill) for wire processing.[15]

Microstructures were evaluated primarily by optical microscopy. Metallographic preparation was routine with a 5-percent Nital solution used to lightly etch (10–50 seconds) the polished surfaces. Observation and photography were carried out on a Ziess Ultraphot III metallograph using Nomarski Differential Interference Contrast (DIC). Indications of the ductility of both hot-rolled and annealed 6.35-mm rod were provided by the drawability of the material.

III. RESULTS AND DISCUSSION

3.1. *Hot-Rolled 6.35-mm (0.25-inch) Rod*

A vertical section through a portion of the Fe-Co-V ternary equilibrium phase diagram (Köster and Schmid)[1] near the nominal composition of Remendur is given in Fig. 3. At 2.7-percent vanadium and temperatures above 950°C the alloy is single-phase FCC (γ). This is the structure of Remendur during hot rolling. The typical micro-

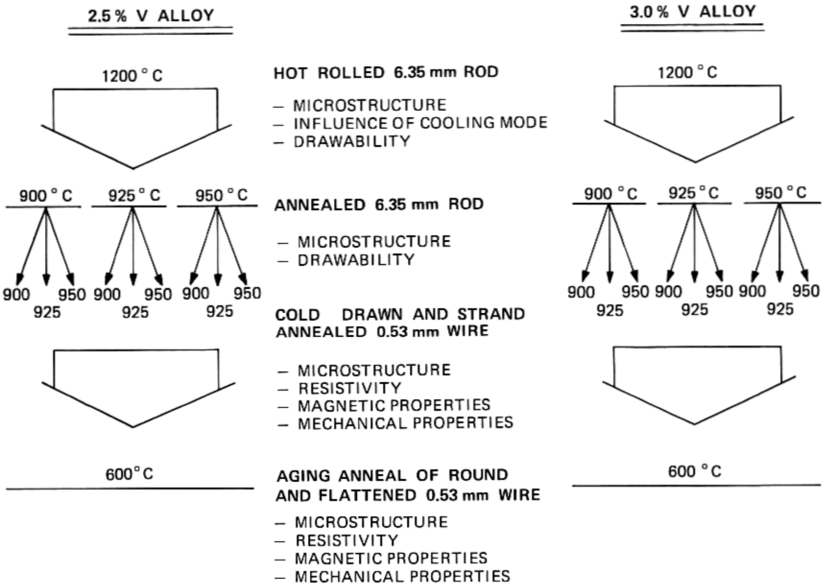

Fig. 2—Synopsis of Remendur processing experiment.

structure developed by cooling from the hot-rolling stage is shown in Fig. 5a. This microstructure is irregular and unconventional. English[6] and Chen[7,8] have amply reviewed the characteristics of this structure and only the most pertinent factors will be restated. Although this composition passes into the two-phase $\alpha_1 + \gamma$ field on cooling, the transformation is sluggish. As a result, with rapid cooling a nonequilibrium single-phase structure which is entirely BCC (α_1), supersaturated in vanadium, is developed (Fig. 5a). No retained γ was detected by X-ray analysis. This has been referred to as a "massive" transformation by English[6] and designated as the α_2 structure. This phase, being a nonequilibrium structure, does not appear on a conventional equilibrium phase diagram. Chen[7,8] proposed that the transformation is actually the more common "martensitic" type. However, the structure of the transformed material is similar for both types[6] and, hence, this will not be discussed here.

The ductility of Remendur with the all α_2 microstructure and as air cooled was tested in a qualitative manner. An attempt was made to draw 6.35-mm rod in this condition (i.e., as hot rolled) into 0.53-mm wire by successive reductions of approximately 20 percent in area. The rod could not survive the first draw pass due to extreme brittleness.

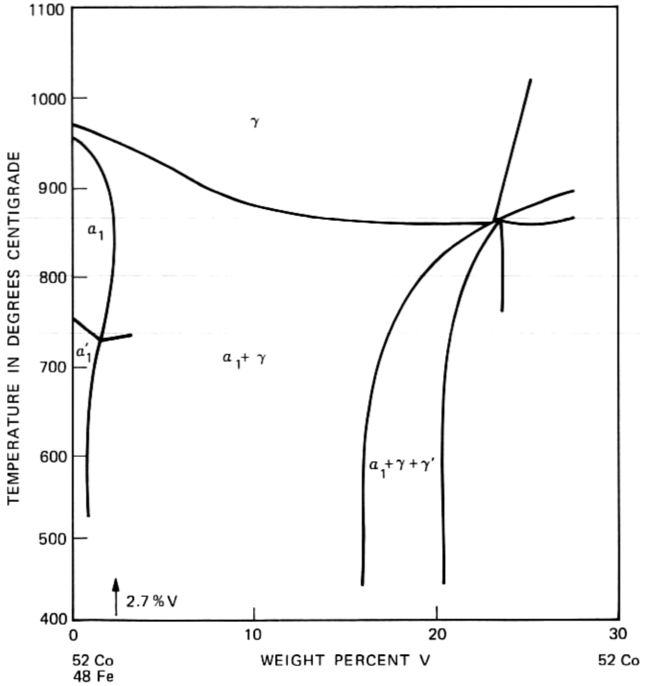

Fig. 3—Vertical section through Fe-Co-V ternary phase diagram, from the work of Köster and Schmid.[1]

3.2 *Annealed 6.35-mm (0.25-inch) Rod*

The brittleness of the air-cooled α_2 microstructure dictates the need for an additional heat treatment to create drawability. A treatment of one-half hour at 925°C (in the two-phase $\alpha_1 + \gamma$ field) followed by an iced brine quench had been found satisfactory in producing cold ductility.[12,15] However, the sensitivity of ductility to this precise time and temperature and the influence of the structure produced by this anneal on subsequent properties developed in the drawn wire had yet to be investigated.

Samples of both the 2.5-percent V and 3.0-percent V alloys in the hot-rolled and air-cooled condition were annealed at 950°C, 925°C, and 900°C for one-half hour and quenched in iced brine. The corresponding microstructures are shown in Figs. 4 and 5. It is apparent that a significant variation in structure occurs as a function of temperature over the relatively small range of 900 to 950°C. At the same annealing temperature, a significant variation as a function of the

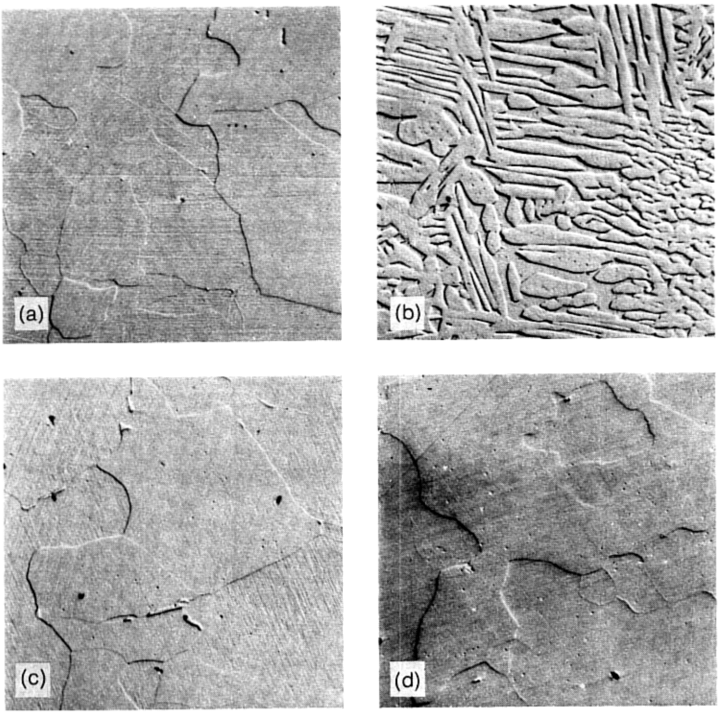

Fig. 4—Quenched microstructures of 2.5% V–6.35-mm Remendur rod in hot-rolled and various annealed conditions; etched; DIC; 600X. (a) Hot-rolled. (b) Annealed; ½ hour; 950°C. (c) Annealed; ½ hour; 925°C. (d) Annealed; ½ hour; 900°C.

small difference in vanadium composition (2.5 to 3.0 percent) is also obvious.

Analysis of the equilibrium phase diagram for this ternary system is necessary to understand the critical nature of temperature and composition for transformations in this range. Based on the vertical sections of the ternary space diagram provided by Köster and Schmid,[1] isotherms (horizontal sections) at temperatures of interest were constructed by the authors. The Fe-Co-rich portions of isotherms at 950, 925, and 900°C are drawn in Fig. 6. The nominal composition of Remendur (2.7-wt. percent V-balance equal Fe/Co) is represented by an X. At all three temperatures the equilibrium structure is in the two-phase $(\alpha_1 + \gamma)$ region, but the amount of each phase can be seen to change radically with temperature. This is a consequence of the γ phase field shrinking rapidly toward the Co corner and the $\alpha_1 + \gamma$ phase field increasing significantly in area as the temperature decreases

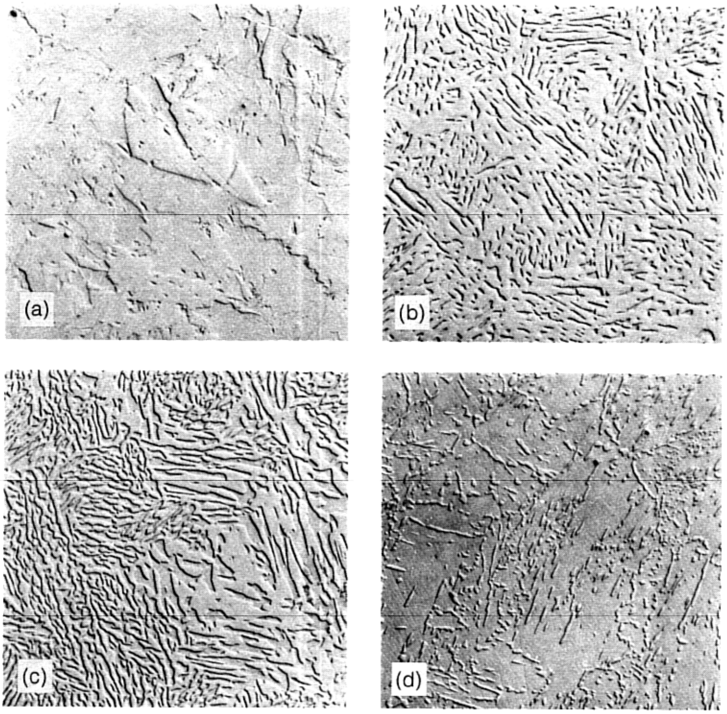

Fig. 5—Quenched microstructures of 3.0% V–6.35-mm Remendur rod in hot-rolled and various annealed conditions; etched; DIC; 600X. (a) Hot-rolled. (b) Annealed; ½ hour; 950°C. (c) Annealed; ½ hour; 925°C. (d) Annealed; ½ hour; 900°C.

from 950°C to 900°C. This α_1 phase (BCC) is an equilibrium solid solution of V in Fe-Co, in contrast to the supersaturated α_2 structure.

The microstructures of Figs. 4 and 5 can be correlated with these diagrams (Fig. 6). At 950°C, the compositions are near the γ phase boundary, hence the two-phase structure is primarily γ with some α_1 (Figs. 4b and 5b).[*,†] The 3.0-percent V material is practically all γ

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[*] The γ phase formed by these anneals transforms to α_2 on quenching identical to the transformation from the single-phase γ region after the hot-rolling stage. Therefore, the two-phase microstructure is actually $\alpha_1 + \alpha_2$ at ambient temperature as photographed. However, the point of interest is the percentage of each phase formed at the elevated temperature which is identical to that existing at ambient for a quenched sample. Thus the designation $\gamma + \alpha_1$ will be maintained for clarity in relating the microstructures to the phase diagrams.

[†] The α_1 etches preferentially to the $\alpha_2(\gamma)$ using the Nital etch. And, under the interference contrast conditions used in all cases, the α_2 phase (raised) appears bright on the top edge as if the light source were shining from the 12-o'clock position. Therefore, for example, in viewing Figs. 4b and 5b the primary or major phase should appear raised, whereas in viewing Fig. 5d the minor phase or particles should appear raised.

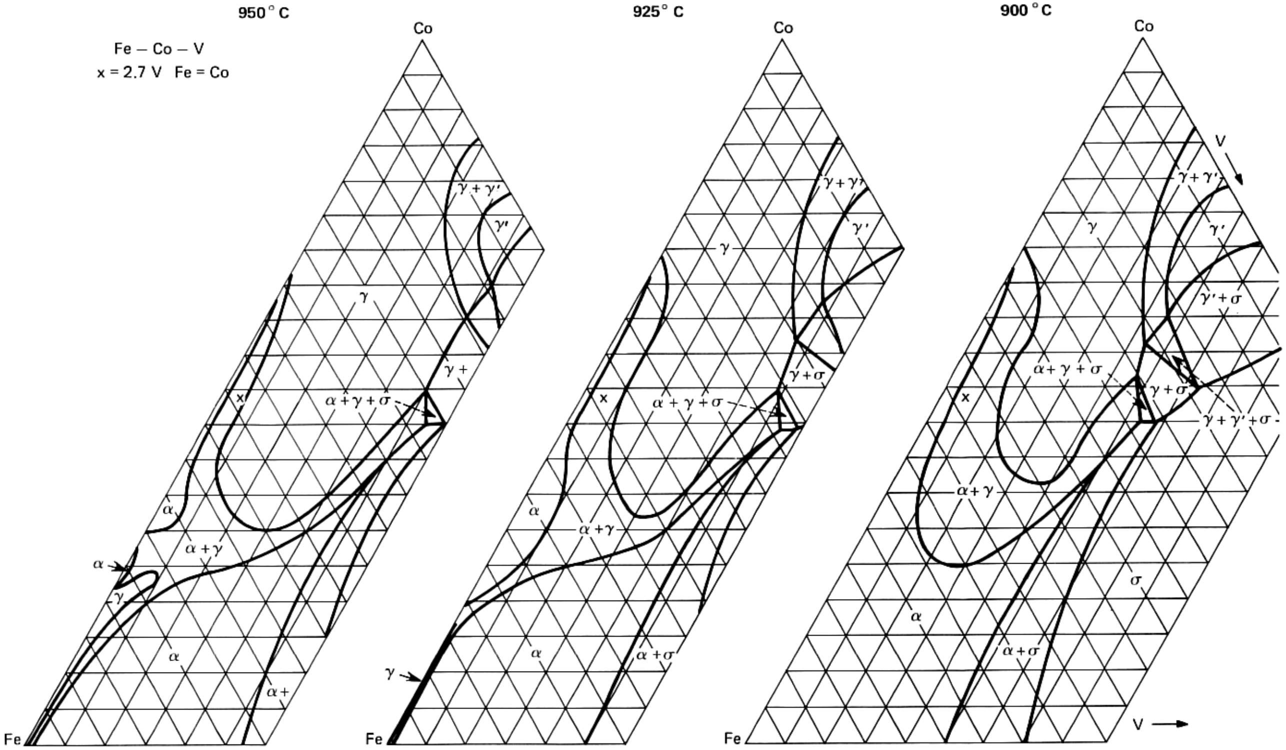

Fig. 6—Isotherms of the Fe-Co-rich portions of the Fe-Co-V ternary equilibrium diagram constructed from the vertical sections in the work of Köster and Schmid.[1]

(Fig. 5b) as this composition almost coincides with the phase boundary. At 900°C, the alloys are near the α_1 phase boundary, hence the two-phase structure is primarily α_1 with some γ (Figs. 4d and 5d). In this instance the lower-vanadium (2.5-percent) alloy is apparently all α_1 (Fig. 4d) as this composition almost coincides with the α_1 phase boundary. For anneals at 925°C, these alloys should have nearly equal proportions of α_1 and γ. This can be seen to be the case for the higher-vanadium alloy (Fig. 5c). However, very little γ is present in the low-vanadium material (Fig. 4c). This may be an indication of a non-equilibrium character in these anneals. Apparently, the more highly strained α_2 lattice of the 3.0-percent V alloy aids sufficiently in the kinetics of nucleation of the γ phase, such that this alloy approaches equilibrium more rapidly than the 2.5-percent V alloy.

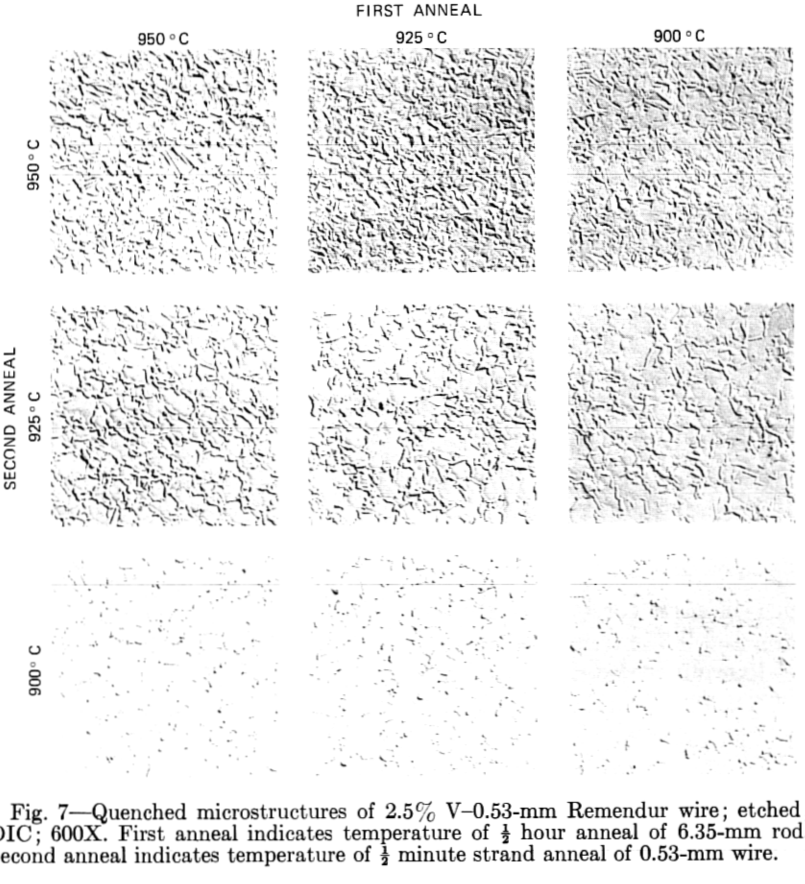

Fig. 7—Quenched microstructures of 2.5% V–0.53-mm Remendur wire; etched; DIC; 600X. First anneal indicates temperature of ½ hour anneal of 6.35-mm rod. Second anneal indicates temperature of ½ minute strand anneal of 0.53-mm wire.

A second influence of the higher vanadium content is apparent in comparing Figs. 4 and 5. A significant degree of grain size refinement occurs for the 3.0-percent V alloy.

All six of the annealed microstructures were found to be ductile. They were drawn from 6.35-mm rod to 0.53-mm wire by successive 20-percent reductions (23 passes) without any failures or evidence of cracking. This result clarifies several points with regard to ductility in Remendur. It has previously been shown that the air-cooled α_2 phase is brittle. English has shown that the α_1 phase orders (α_1') on other than very rapid rates of cooling[9] and alludes to the enhanced brittleness of this microconstituent.[6] Stoloff and Davies presented more complete evidence of the significant loss of ductility due to ordering.[11] Chen[8] and Davies[16] have shown that some degree of ductility also can be created in the α_2 constituent by the rapid cooling provided by a quench. However, Chen[8] demonstrated that the quenched single-phase α_2 structure does not provide the degree of ductility of the quenched two-phase structure. The influence of the quench on the α_2 constituent may also be related to the ordering phenomena although this has not yet been verified. Thus the two-phase structures developed by anneals between 900 and 950°C and followed by a quench are believed to provide the maximum degree of ductility attainable in this alloy. Anneals at temperatures below 900°C but above the ordering temperature ($\approx$ 720°C) followed by a quench may also produce a ductile structure. However, structures produced by lower-temperature anneals for drawability may be undesirable for developing proper magnetic properties in the final reed.

3.3 *Annealed 0.53-mm (0.021-inch) Wire*

Remendur rods of both compositions and all three annealing temperatures were drawn directly to 0.53-mm wire (6 samples). Sections of each sample were strand-annealed at furnace set temperatures of 900, 925, and 950°C for 30 seconds to produce a series of 18 samples with different combinations of composition, 6.35-mm anneal temperature, and 0.53-mm anneal temperature. The actual peak temperatures as determined by drawing a thermocouple through the furnace were 935°C, 913°C, and 890°C. The appropriate microstructures are shown in Figs. 7 and 8. The most significant points to be noted are as follows:

(*i*) For the *low*-vanadium alloy (Fig. 7) the second anneal uniquely determines the percentages of α_1 and γ. (Compare the identical microstructures of each horizontal row.) This is due to the fact that, con-

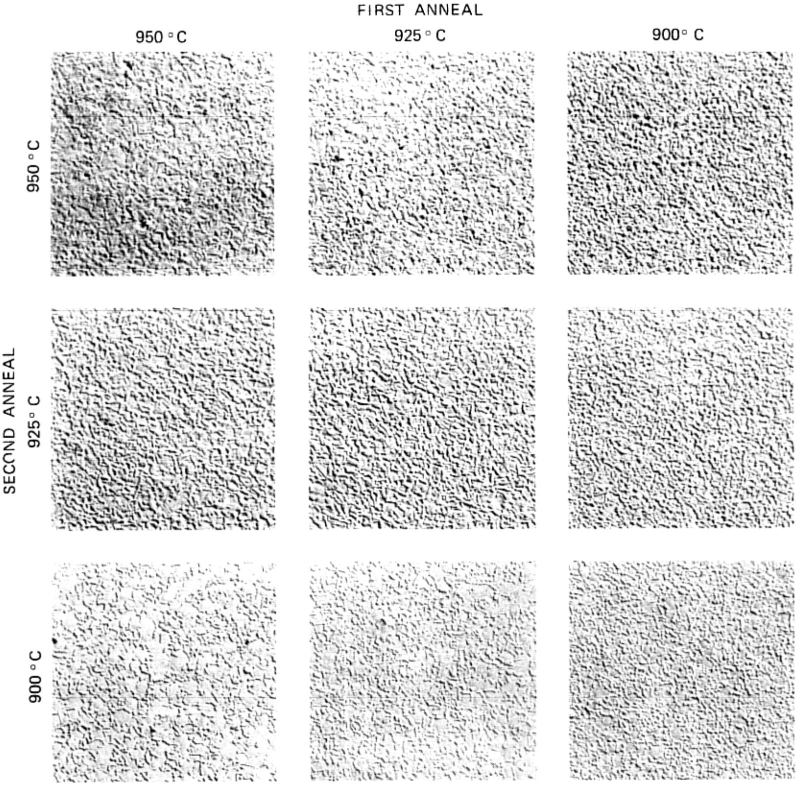

Fig. 8—Quenched microstructures of 3.0% V–0.53-mm Remendur wire; etched; DIC; 600X. First anneal indicates temperature of ½ hour anneal of 6.35-mm rod. Second anneal indicates temperature of ½ minute strand anneal of 0.53-mm wire.

trary to the case for the anneals of the 6.35-mm rod, the anneals in the 0.53-mm wire apparently produce a nearly equilibrium two-phase structure. This is likely a consequence of the aid to kinetics of nucleation provided by the deformation of wire drawing.

(*ii*) For the *high*-vanadium alloy (Fig. 8) the second anneal also primarily determines the percentages of $\alpha_1 + \gamma$ in qualitative agreement with the equilibrium diagrams (Fig. 6). The first anneals have little effect on phase percentages in the annealed wires indicating the 0.53-mm, 30-second strand anneals *may* produce equilibrium structures.

(*iii*) The same refinement in grain size of the high-V alloy compared to the low-V alloy previously noted is again present. An additional and substantial refinement occurs in the reduction of the wire from 6.35 to 0.53 mm (compare Figs. 5 and 8).

3.4. *600°C-Annealed 0.53-mm Wire and 0.18-mm Ribbon*

Each of the 18 samples was cut into two lengths. One length was rolled to a thickness of 0.18 mm (0.007 inch) and the other length remained as 0.53-mm wire. These conditions simulate the paddle and shank, respectively, of a typical reed. All 36 samples were given the standard 600°C, two-hour anneal in dry H_2 which is used to produce a square magnetic hysteresis loop with a specified range of coercive force.

The values of the coercive force developed for the various combinations of strand-annealing temperature and vanadium content in the round and flat sections are listed in Table II, and representative microstructures for both round and flattened sections are shown in Figs. 9 and 10. The details of the phase transformation which occurs during this 600°C anneal are presented in another paper[17] and will not be restated here. It is sufficient to note that the final microstructure developed in the round (undeformed) wire by this anneal is a strong

Table II—Coercive Force in Oersteds of 0.53-mm Round Wire and 0.18-mm Flat Ribbon in the 600°C–Annealed Condition

(2.5 wt. % V)
First Anneal

		950°C		925°C		900°C	
		F	R	F	R	F	R
Second Anneal	950°C	22.9	11.9	23.7	11.9	24.1	11.5
	925°C	21.8	11.0	22.9	11.5	23.7	11.5
	900°C	22.6	11.9	22.9	8.3	23.3	5.1

(3.0 wt. % V)
First Anneal

		950°C		925°C		900°C	
		F	R	F	R	F	R
Second Anneal	950°C	29.7	18.2	30.8	20.2	30.8	20.6
	925°C	30.8	23.7	32.0	24.9	32.0	24.6
	900°C	30.5	17.0	30.8	17.8	31.3	21.0

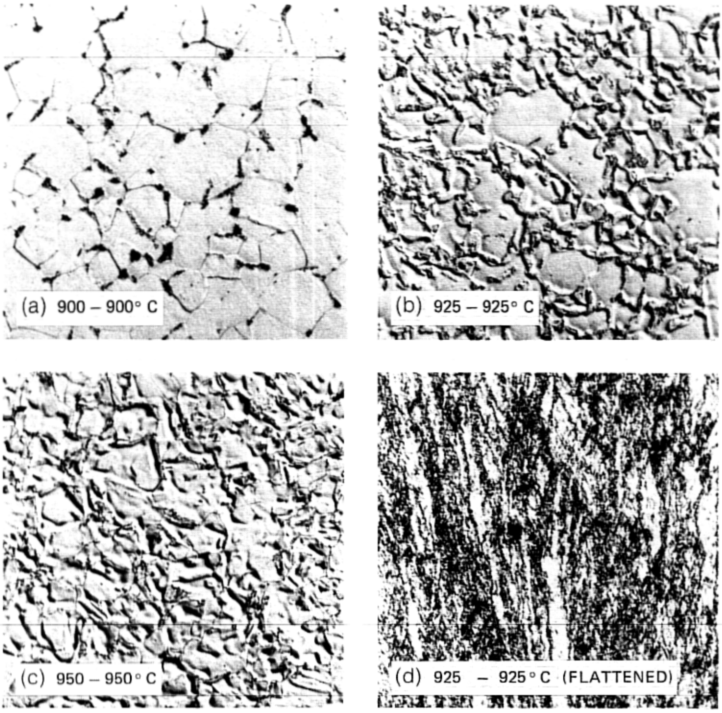

Fig. 9—Microstructures of 0.53-mm round wire and 0.18-mm flattened ribbon following 600°C, 2-hour anneal of 2.5% V alloy; etched; DIC; 1500X. Subcaptions specify temperatures of first and second anneals prior to 600°C anneal.

function of the microstructure from the strand-annealed state. This may be seen by comparing Fig. 7 to Fig. 9 and Fig. 8 to Fig. 10 for the low- and high-vanadium alloys, respectively. For the low-V alloy strand annealed at 900°C (Fig. 9a) the large grain α_1' matrix with particles of γ at the grain boundaries produces a coercive force of only 5.1 oersteds. The other structures show a coercive force of approximately 11.0 oersteds with a maximum value of only 11.9 oersteds. This demonstrates the significant influence of vanadium content on coercivity and the need to keep the minimum permissible vanadium content above the 2.5-wt. percent level to achieve the required minimum value of 18 oersteds on the round section of the reed for remreed applications.

The high-V alloy shows a clear correlation between magnetic properties after the 600°C anneal and the prior strand-annealed microstructure. As shown in Table II a peak in coercivity of the round wire is attained for the 925°C strand anneal of 0.53-mm wire. This is the

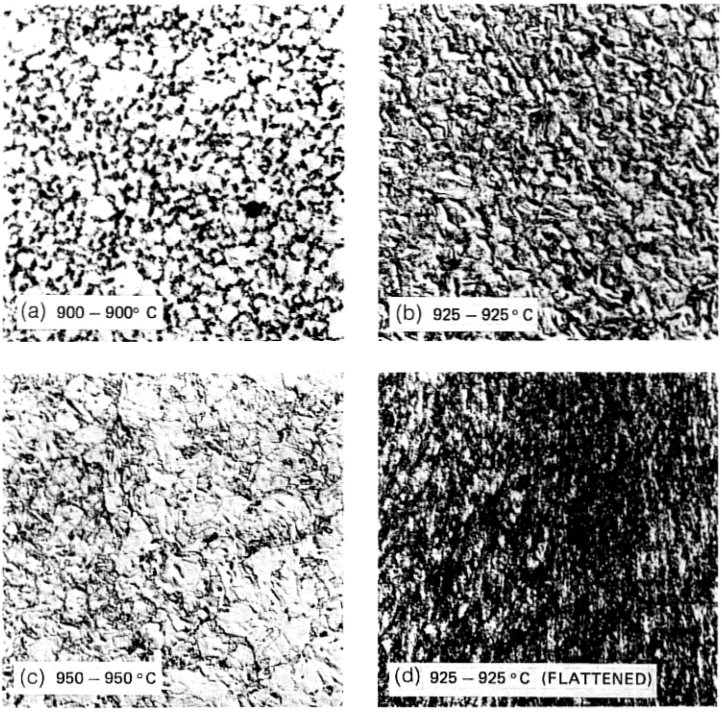

Fig. 10—Microstructures of 0.53-mm round wire and 0.18-mm flattened ribbon following 600°C, 2-hour anneal of 3.0% V alloy; etched; DIC; 1500X. Subcaptions specify temperatures of first and second anneals prior to 600°C anneal.

state of a nearly equal distribution of the two-phase $\alpha_1 + \alpha_2$ structure (Fig. 8) which produces a more uniform distribution of α_1' (ordered α_1) and γ (Fig. 10b) during the subsequent 600°C anneal. Coercivity decreases for strand anneals at higher or lower temperatures where the α_1 or α_2 phase predominates.

The flattening operation eliminates most of the microstructural influence on final magnetic properties as shown in Table II. For the high-V alloy, all values are in the range of 30 to 32 oersteds and for the low-V alloy in the range of 22 to 24 oersteds. All microstructures are similar to that shown in Figs. 9d and 10d which consists of the very fine distribution of γ in an α_1' matrix.

IV. CONCLUSIONS

The primary conclusion from this study is that the microstructure of 2–3-wt. percent vanadium Remendur is extremely sensitive to annealing temperature and vanadium content. Magnetic characteristics of Remendur wire and the remreed contact and the mechanical properties, particularly drawability, of the material are dependent on the precise microstructure developed during processing. More specific conclusions are listed as follows.

(*i*) Anneals in the range of 900 to 950°C produce a two-phase α_1 (BCC) + γ (FCC) structure which changes to a two-phase α_1 (BCC) $+\alpha_2$ (supersaturated BCC) structure on rapid cooling. The percentages of α_1 and γ formed are a strong function of temperature with increasing temperatures yielding greater percentages of γ (α_2).

(*ii*) The one-half-hour anneal of low-vanadium 6.35-mm rod does not produce the equilibrium two-phase structure defined by the phase diagram. Increased vanadium content assists in producing a nearly equilibrium structure as does the deformation of wire drawing for the anneal of 0.53-mm high- and low-vanadium wire.

(*iii*) The percentages of α_1 and γ (α_2) formed in the 0.53-mm wire are primarily a function of the temperature of the anneal. The temperature of the 6.35-mm rod anneal has little influence on determining these final percentages.

(*iv*) The all α_2 structure created by slow to moderate rates of cooling from the γ phase region is brittle and cannot be cold drawn. However, a two-phase mixture of $\alpha_1 + \alpha_2$, developed by quenching from anneals in the range of 900 to 950°C (the two-phase $\alpha + \gamma$ region), is sufficiently ductile to be drawn from 6.35-mm rod directly to 0.53-mm wire. If the rate of cooling is not sufficiently rapid from the 900 to 950°C range, the α_1 structure orders[9] to form α_1' which has also been shown to be brittle and, therefore, nondrawable to wire.[11]

(*v*) Increased vanadium content and the deformation of cold drawing both are influential in refining the grain size of the two-phase Remendur structure. The refined grain size and higher vanadium content influence the magnetic properties of 0.53-mm Remendur wire which has been annealed at 600°C for 2 hours by increasing the coercivity.

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