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THESIS

AN ELECTRON MICROSCOPY STUDY OF TWEED
MICROSTRUCTURES AND PREMARTENSITIC EFFECTS
IN HIGH DAMPING 53Cu45Mn2Al ALLOY

by

Larry Leroy Mayes

March 1988

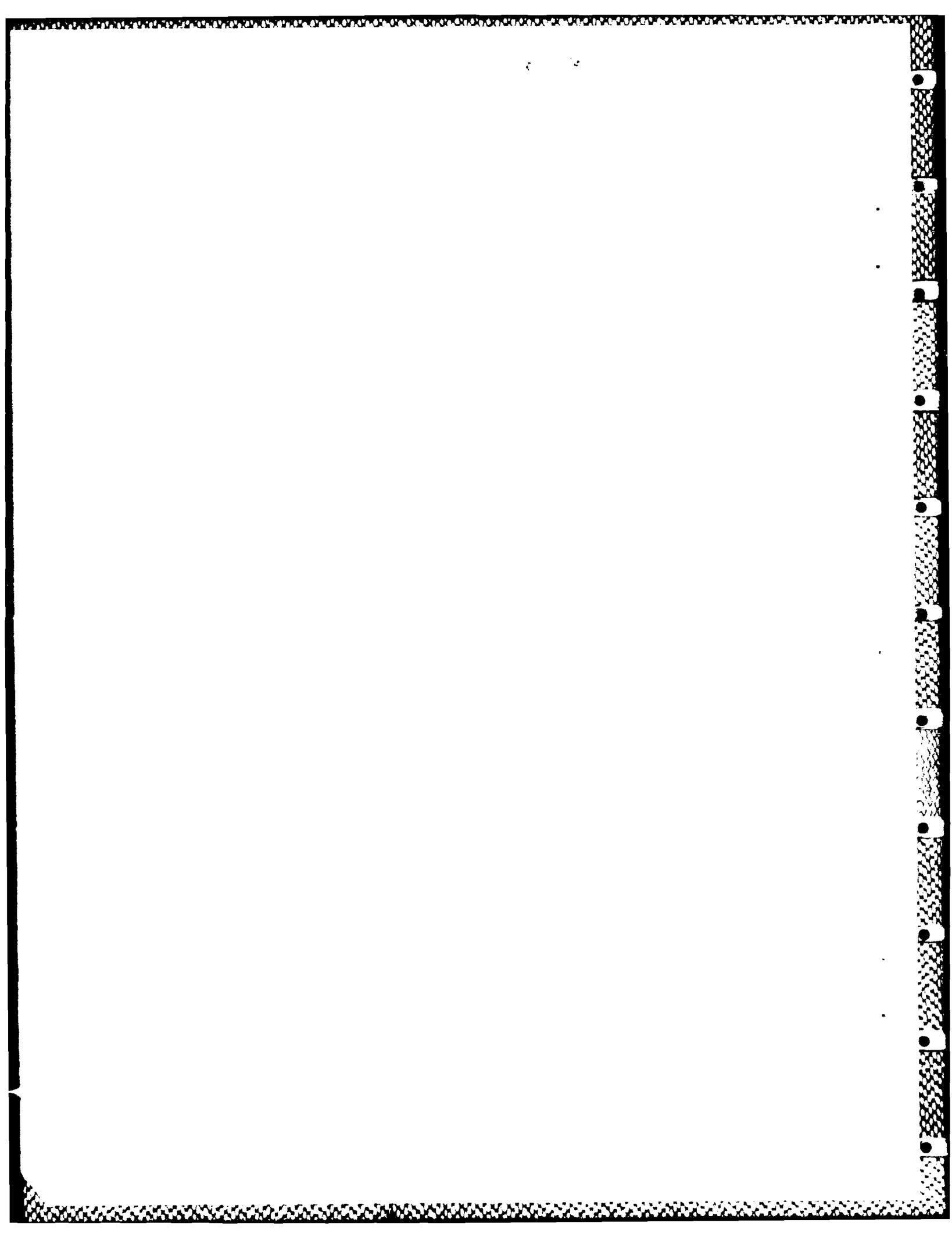
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REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED		1b. RESTRICTIVE MARKINGS NONE	
2a. SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for Public Release; Distribution is Unlimited.	
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE			
4. PERFORMING ORGANIZATION REPORT NUMBER(S)		5. MONITORING ORGANIZATION REPORT NUMBER(S)	
6a. NAME OF PERFORMING ORGANIZATION Naval Postgraduate School	6b. OFFICE SYMBOL (If applicable) 69	7a. NAME OF MONITORING ORGANIZATION Naval Postgraduate School	
6c. ADDRESS (City, State, and ZIP Code) Monterey, California 93943-5000		7b. ADDRESS (City, State, and ZIP Code) Monterey, California 93943-5000	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER	
8c. ADDRESS (City, State, and ZIP Code)		10. SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO.	PROJECT NO.
		TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) AN ELECTRON MICROSCOPY STUDY OF TWEED MICROSTRUCTURES AND PREMARTENSITIC EFFECTS IN HIGH DAMPING 53Cu45Mn2Al ALLOY			
12. PERSONAL AUTHOR(S) MAYES, Larry L.			
13a. TYPE OF REPORT Master's Thesis	13b. TIME COVERED FROM TO	14. DATE OF REPORT (Year, Month, Day) March 1988	15. PAGE COUNT 62
16. SUPPLEMENTARY NOTATION The views expressed in this thesis are those of the author and do not reflect the official policy or position of the Department of Defense or the U.S. Government.			
17. COSATI CODES FIELD GROUP SUB-GROUP		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) High Damping, Strain Dependent Damping, INCRAMUTE, (Cu-Mn Alloys, Premartensitic Effects, Flickering, Tweed Microstructures, Manganese Alloys, Thesis, etc.)	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The evolution of tweed microstructure upon aging a 53Cu45Mn2Al alloy was studied in the transmission electron microscope (TEM). Characteristic (110) tweed contrast develops after the alloy is aged for 4 hours or longer at 400 C, which apparently is just within the miscibility gap, and is then cooled to room temperature. The microstructure evolved is proposed to consist of a small proportion of Mn-rich regions in a Cu-rich matrix. As the phase separation proceeds, the Neel temperature and the FCC-to-FCT transition temperature both rise within these Mn-rich domains, and as a result they begin to display incipient lattice instabilities when observed at room temperature. This takes the form of a "flickering" effect in the TEM image. The flickering consists of consistent and repetitive contrast variations at specific locations in the microstructure. The significance of these microstructural features to damping is discussed.			
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED	
22a. NAME OF RESPONSIBLE INDIVIDUAL Prof Jeff Perkins		22b. TELEPHONE (Include Area Code) (408) 646-2216	22c. OFFICE SYMBOL 69Ps

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An Electron Microscopy Study of Tweed Microstructures
and Premartensitic Effects in High Damping
53Cu45Mn2Al Alloy

by

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Submitted in partial fulfillment of the
requirements for the degree of

MASTER OF SCIENCE IN MATERIAL SCIENCE

from the

NAVAL POSTGRADUATE SCHOOL
March 1988

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ABSTRACT

The evolution of tweed microstructure upon aging a 53Cu45Mn2Al alloy was studied in the transmission electron microscope (TEM). Characteristic {110} tweed contrast develops after the alloy is aged for 4 hours or longer at 400 C, which apparently is just within the miscibility gap, and is then cooled to room temperature. The microstructure evolved is proposed to consist of a small proportion of Mn-rich regions in a Cu-rich matrix. As the phase separation proceeds, the Neel temperature and the FCC-to-FCT transition temperature both rise within these Mn-rich domains, and as a result they begin to display incipient lattice instabilities when observed at room temperature. This takes the form of a "flickering" effect in the TEM image. The flickering consists of consistent and repetitive contrast variations at specific locations in the microstructure. The significance of these microstructural features to damping is discussed.

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ACKNOWLEDGEMENT

A special thanks to Professor Jeff Perkins for his guidance, the invaluable long talks on the subject and his extreme patience with this slow-to-learn student. I would also like to thank Professor Tadayoshi Yamashita for his generous assistance in developing thin foil procedures and analyzing the TEM photographs. Finally, I want to thank my wife, Linda, and my daughters, Cynthia, Staci and Jaime, without whose support and patience this research could not have been accomplished.

I. INTRODUCTION

A. GENERAL

The reduction of noise and vibration is of critical interest to the U. S. Navy. This is because noise and vibration can lead to several undesirable effects, including:

- * Potential hearing loss and/or impaired performance of personnel.
- * Reduced performance or misalignment of critical electronics and other equipment.
- * Shortened fatigue life of equipment.
- * Increased detection ranges of surface ships and submarines.

Three possible practical approaches for noise and vibration control are:

1. Isolation - the reduction of energy transmission between the sources (moving parts) and the surface that can radiate the energy.
2. Structural dissipation - attenuation of the energy somewhere in the structure by the use of isolation pads.
3. Material dissipation - components are made of alloys or composites which have high damping capacity.

The first two of these, isolation and structural dissipation, are the approaches generally used in current engineering practice. Although these methods are effective, they have their drawbacks (e.g., the increased weight and space necessary to accommodate the materials used for isolation or dissipation).

Therefore, this thesis is part of an ongoing research program at the Naval Postgraduate School to determine the damping characteristics of existing commercial alloys and to discover the microstructural mechanisms of damping. An understanding of these mechanisms should contribute to an ability to select alloys with the potential for high damping, or even to design of alloys from their basic properties.

B. BACKGROUND

Any system possessing mass and elasticity is capable of vibration. Damping is the property of a material which describes how rapidly these vibrations decay once excited within the material. All materials exhibit some degree of vibrational damping, but most structural alloys exhibit poor damping capacity at the amplitude of stress associated with vibration and noise emission. The specific damping capacity (SDC) of most structural materials is quite low (i.e. less than 1%). A summary of SDC, yield strength and density is provided in Table 1 for selected structural materials and some high damping alloys. Gray cast iron, which has been considered an unusually effective energy absorbing structural metallic material, actually has a SDC value in the range of 5% to 10%. However, several high damping "quiet" alloys have damping capacities of 25% to 50%.

Although several alloy systems have been shown to exhibit high damping capacity, each of these alloy systems has demonstrated some drawback. For example:

- * High cost due to the limited production of required base materials for alloy production.
- * Poor resistance to corrosion in a marine environment.
- * Aging effects (reduced damping with time) at the desired operating conditions.
- * Difficulty in machining or casting the alloy.
- * Difficulty in preserving the damping capacity while forming the required product.

Consequently, research is being done to design alloys that eliminate some or all of these drawbacks.

When considering the mechanisms of damping in materials, the basic idea is that in order to have damping, some features in the microstructure have to move. Some examples of moveable features are:

TABLE 1
 DAMPING CHARACTERISTICS OF SELECTED METALS
 AT ROOM TEMPERATURE [Ref. 1: p. 203]

Metal	SDC (%)	Yield Strength (10 ³ psi)	Density (gm/cm ³)
Magnesium (wrought)	49	26	1.74
Cu-Mn alloys (Ingramute, Sonoston)	40	45	7.5
Ni-Ti alloy (Nitinol)	40	25	6.45
Fe-Cr-Al alloy (Silentalloy)	40	40	7.4
High-C gray iron	19	25	7.7
Nickel (pure)	18	9	8.9
Iron (pure)	16	10	7.86
Martensitic stainless steel	8	85	7.7
Gray cast iron	6	25	7.8
SAP (aluminum powder)	5	20	2.55
Low-carbon steel	4	50	7.86
Ferritic stainless steel	3	45	7.75
Malleable, modular cast irons	2	50	7.8
Medium-carbon steels	1	60	7.86
Austenitic stainless steel	1	35	7.8
1100 Aluminum	0.3	5	2.71
Aluminum alloy 2024-T4	<0.2	47	2.77
Nickel-based superalloys	<0.2	Range	8.5
Titanium alloys	<0.2	Range	4.5
Brasses, bronzes	<0.2	Range	8.5

- * Atoms (e.g., stress induced movement of carbon or nitrogen in iron).
- * Point Defects.
- * Dislocations.
- * Grain Boundaries.
- * Twin boundaries.
- * Magnetic domain boundaries.
- * Interphase boundaries.

While all of these features provide some damping, some are better than others. Twin boundaries, magnetic domain boundaries and interphase boundaries seem to provide the best damping mechanisms. Certain variables of the imposed vibration, such as frequency, strain amplitude and number of cycles, can also influence the degree of damping [Ref. 2].

1. Metallurgy of the Cu-Mn Alloy System

The present research was focused on the characteristics of a specific high damping alloy, 53Cu-45Mn-2Al (weight %). It was previously shown [Ref. 3] that this alloy develops a "tweed" microstructure after aging. However, the process of microstructural evolution of the tweed and the exact mechanisms of damping in this alloy have not been determined. It was to these questions that the present research was addressed.

A brief review of some of the features of the Cu-Mn alloy system, as well as the possible origins of a tweed type microstructure, will be presented in order to introduce the potential microstructural damping mechanisms.

The Cu-Mn binary alloy system displays a broad single-phase region at elevated temperature [Ref. 4]. This FCC gamma (γ) phase is shown in the Cu-Mn phase diagram, Figure 1.1. When an FCC γ -phase alloy is rapidly cooled, the alloy first undergoes an antiferromagnetic ordering [Refs. 5,6,7,8,9,10]. The initiation of antiferromagnetism is associated with a concentration dependent temperature, the Neel temperature (T_N), which increases with

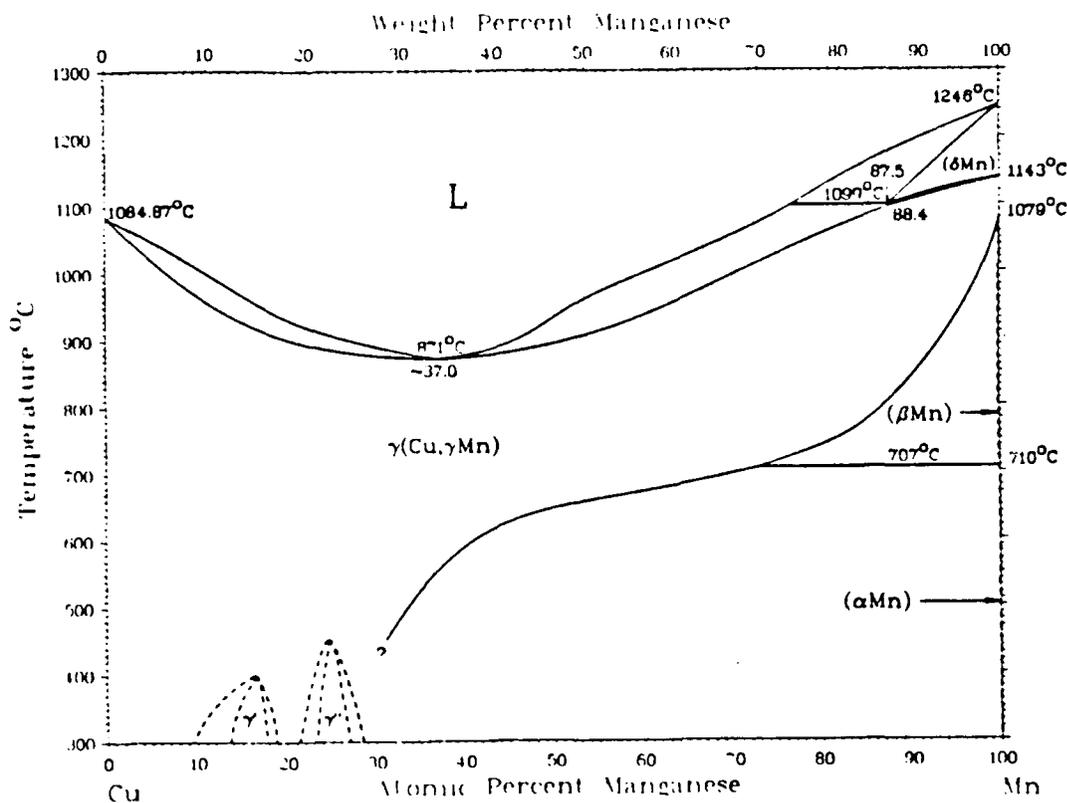


Figure 1.1 Phase Diagram of Copper-Manganese Binary System.

increasing Mn content and has been carefully determined by previous workers [Refs. 6,10]. At this critical temperature the atomic magnetic dipoles of adjacent Mn atoms become aligned in an antiparallel manner. Domains are formed with mismatches of the antiferromagnetic alignment at the domain boundaries [Ref. 11: p. 473]. This arrangement results in zero net magnetism. Figure 1.2 shows the relationship between T_N and Mn content.

Closely coincident with the antiferromagnetic ordering, the alloy transforms from the FCC γ -phase to a metastable twinned FCT structure [Refs. 6,12,13,14,15] which may be regarded as quasi-martensitic (that is, a shear transformation with very low lattice strain). The temperature of the FCC-to-FCT transition for solution heat treated and quenched alloys, which may be called the martensite start (M_s) temperature, varies approximately linearly with Mn

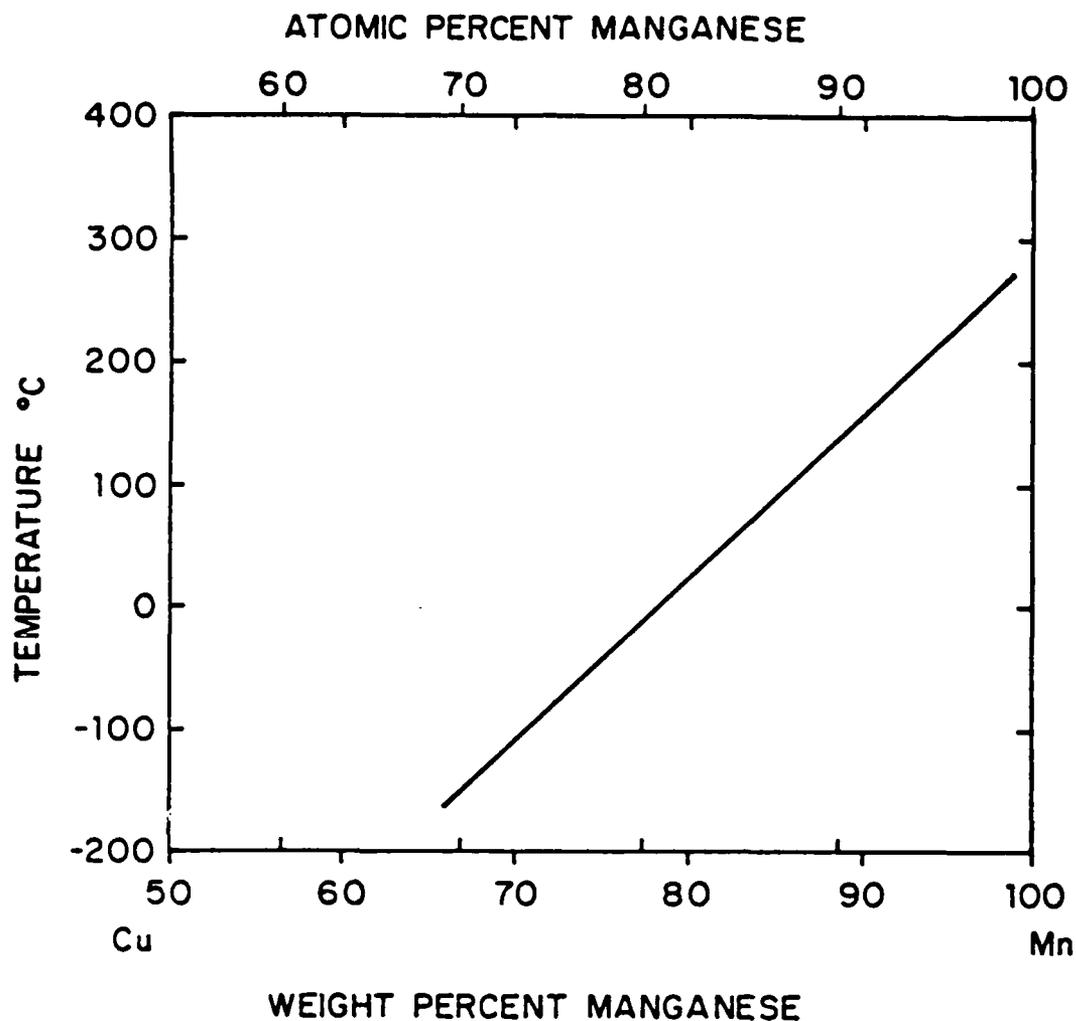


Figure 1.2 Neel Temperature (T_N) vs. Mn Concentration in As-quenched Alloys [Ref. 16].

content. The M_s temperature increases with increasing Mn content [Refs. 5,6,12], as is shown on Figure 1.3.

As a result of the composition-dependence of T_N and M_s , the transition temperature for the FCC-to-FCT transformation falls below room temperature when the Mn content is reduced to about 82% Mn. Therefore, quenched alloys of <82% Mn, which have been solution heat treated in the γ -single

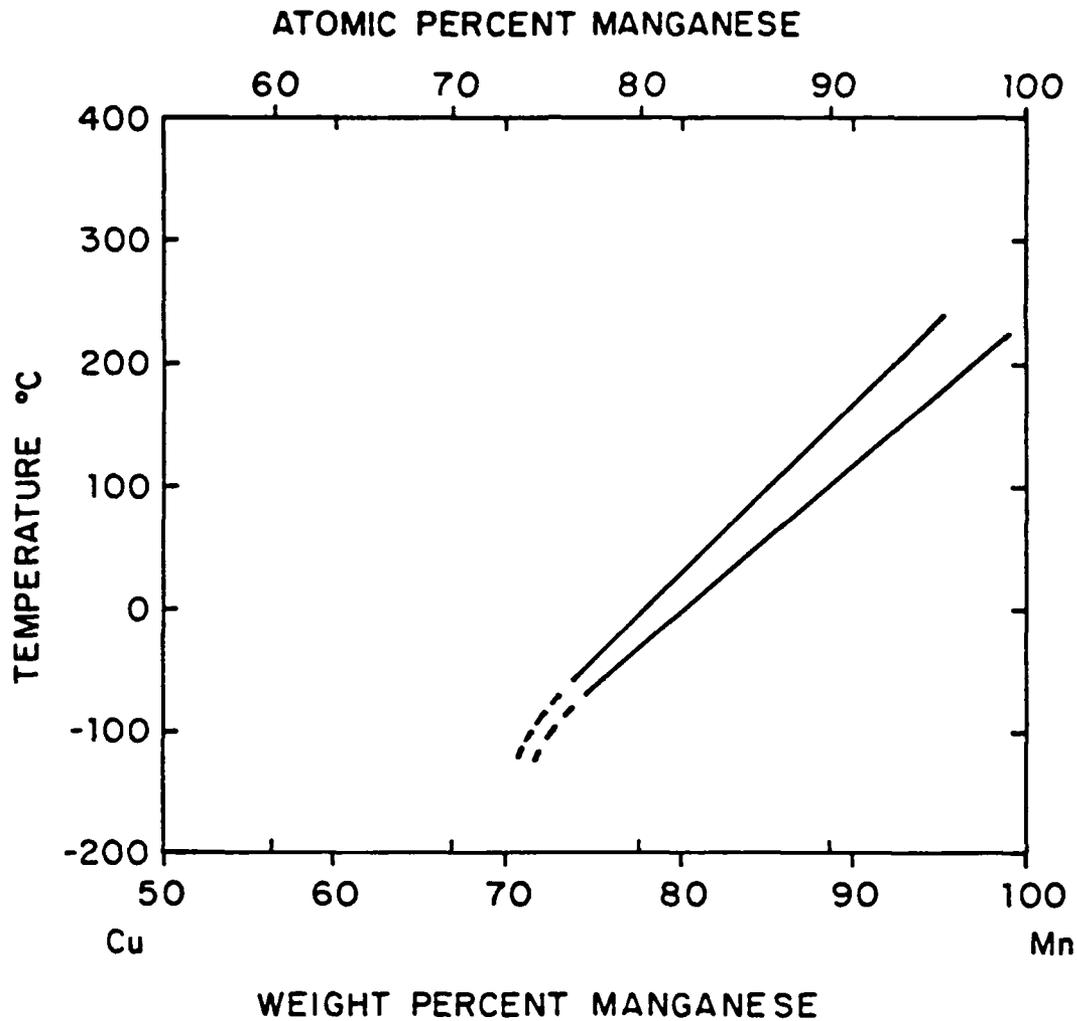


Figure 1.3 The FCC-to-FCT Transformation in Cu-Mn Alloys [Ref. 12].

phase region, remain as FCC at room temperature. For alloys >82% Mn, the FCC γ -phase transforms to the twinned FCT structure.

Another important characteristic of Cu-Mn alloys is the existence of a miscibility gap, although there is some uncertainty about its exact location [Refs. 16,17,18]. This miscibility gap is shown on Figure 1.4, along with the plot of the T_N and M_s temperatures. When held within the

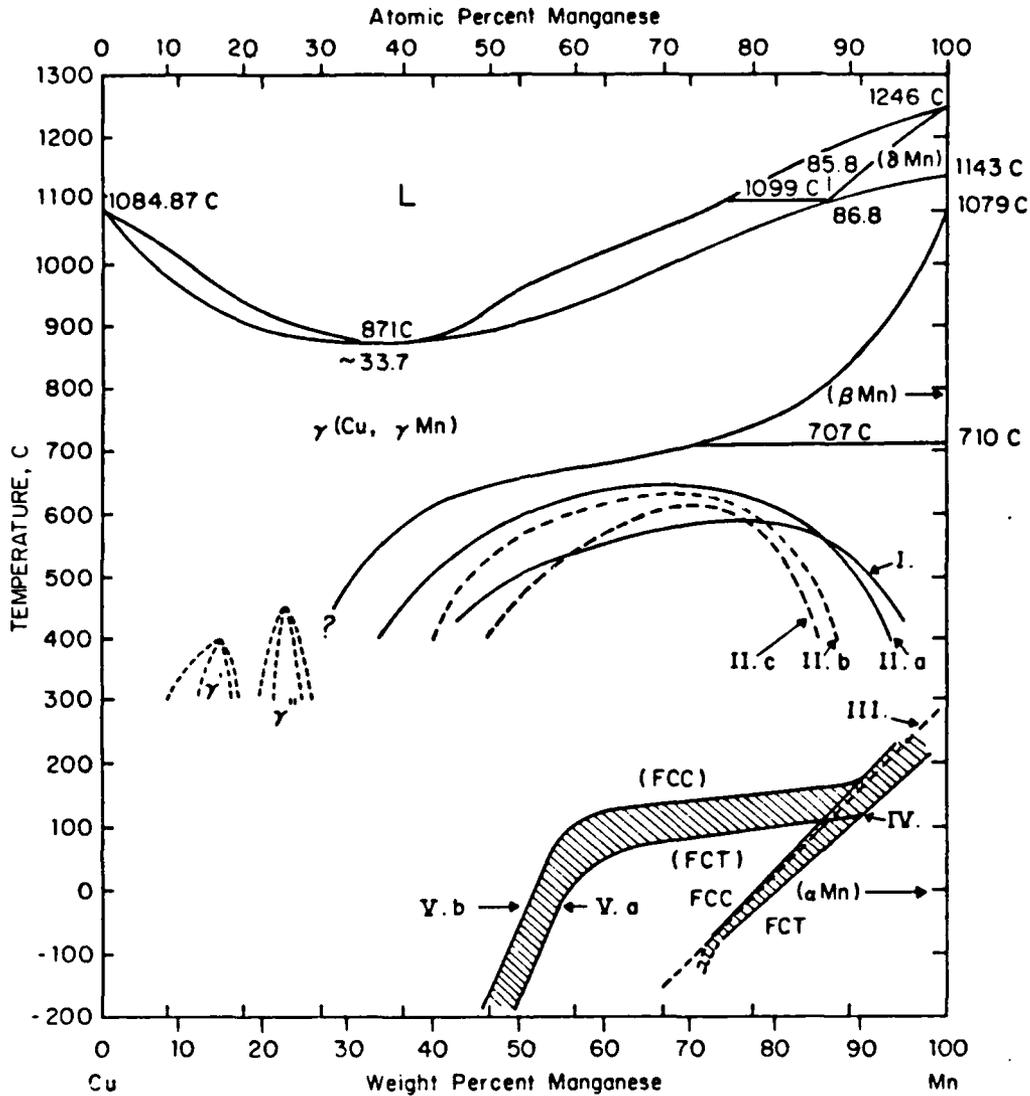


Figure 1.4 Phase Diagram of Cu-Mn binary system with Miscibility Gap, M_s , and T_N .

- I. Miscibility gap [Ref.16].
- II. Miscibility gap [Ref. 19].
 - a. Layering range.
 - b. Chemical spinodal.
 - c. Coherent spinodal.
- III. Neel temperature [Ref. 16].
- IV. FCC-to-FCT transition in quenched alloy [Ref. 12].
- V. FCC-to-FCT transition in aged alloy [Ref. 20].

miscibility gap, an alloy separates into Mn-rich and Cu-rich regions [Refs. 5,19,21,22]. Although equilibrium α -Mn precipitates will ultimately form, the effects of interest occur at relatively early aging times where a fine-scale composition modulation initially develops. This apparently creates localized Mn-rich regions wherein antiferromagnetic ordering and quasi-martensite formation may occur when quenched to room temperature. These transitions can occur because T_N and the M_s temperature are effectively raised within these Mn-rich regions, so that quenching to room temperature may give rise to the FCC-to-FCT transformation. Thus, alloys between 50% and 82% Mn may transform from FCC-to-FCT upon quenching to room temperature after aging for various times between 300 C to 600 C [Refs. 20,21,23]. This is explained by the existence of the miscibility gap which leads to decomposition of the FCC phase upon aging.

Several workers [Refs. 3,20] have concluded, from x-ray investigation, that the FCC-to-FCT transformation does not occur for alloys of <50 at. % (approximately 46.4 wt. %) Mn. For example, Reskusich and Perkins [Ref. 3] demonstrated that the 53Cu-45Mn-2Al alloy lacked indication of the FCC-to-FCT transformation, but presented a tweed appearance as damping increased to a maximum and then decreased.

2. Damping Mechanisms in Cu-Mn alloys

In unaged Mn rich Cu-Mn alloys, a twinned FCT microstructure is believed to be the source of damping [Refs. 21,23,24,25]. Hedley [Ref. 21] stated that the energy absorption takes place by at least two mechanisms:

1. A frictional term due to the microtwin boundary moving through the lattice.
2. The energy involved in the reversible rotation of magnetic moments associated with each Mn ion.

There are at least two other mechanisms that should be considered in more detail, and which formed a focus of the present research. One of these is the role of the pre-martensitic tweed structure. The other is the possible

importance of the FCC-to-FCT transformation as a result of stress-induction under vibrational conditions.

3. Tweed Structure

The term "tweed" is a generic term for a particular sort of diffraction contrast in TEM images. This typically consists of irregular lines of contrast lying approximately along traces of $\{110\}$ planes of a cubic parent phase, forming a kind of cross-hatched pattern. The lines of contrast obey extinction rules which are consistent with them being due to $\langle 110 \rangle$ shear distortions of the $\{110\}$ planes [Refs. 26,27].

There are numerous systems in which tweed microstructures are observed, and a variety of origins of the shear distortions which are one of the two basic conditions that must be satisfied for this type of contrast to develop. The conditions are:

1. A source of finely distributed centers of asymmetric strain.
2. An elastically anisotropic matrix phase.

In different systems the strain centers may be:

- * G-P zones [Ref. 26].
- * Fine precipitates [Ref. 28].
- * Ordered domains [Ref. 29].
- * Domains in which there is an incipient lattice transition which distorts the lattice [Ref. 30].

For many cubic lattices, the existence of certain soft elastic constants often provides a matrix phase which is particularly susceptible to shear distortions of the type $\{110\}\langle 110 \rangle$ [Ref. 31]. In order to excite this distortion, based on the $\langle 110 \rangle$ transverse phonon mode, the straining centers distributed in the matrix must be asymmetric. If they are symmetric, they are more likely to excite the typical soft $\langle 100 \rangle$ longitudinal mode, the distortions of which lead to $\{100\}$ contrast traces rather than the typical $\{110\}$ contrast traces of a true tweed microstructure [Ref. 32].

The "sharpness" of a tweed microstructure, that is to say the degree of alignment and contrast, is therefore dependent on the distribution, the nature and the magnitude of the strain centers, as well as the degree of anisotropy of the matrix phase. If any of these factors is deficient (i.e. not enough strain centers, not enough asymmetric distortion provided by each strain center, or insufficient elastic anisotropy of the matrix), the typical $\{110\}$ aligned tweed contrast will not be observed. In these "weak" cases the image will simply present a "mottled" contrast. Therefore, many "tweedy" alloys show a variation in the distinction of the tweed contrast as the distribution and strength of the strain centers changes, for example, with aging [Ref. 26], or upon cooling toward a temperature range of lattice instability [Ref. 30].

Cu-Mn alloys have been previously reported to display tweed contrast microstructures under certain conditions of heat treatment and observation temperature [Refs. 15,16,23,33,34]. The present work with the 53Cu-45Mn-2Al alloy provided a unique situation with regard to the development of the tweed contrast, in that the response of the lattice could be controlled quite closely by the aging treatment. Also, subsequent changes on cooling pertained to just a certain proportion of the microstructure, namely that proportion which had achieved a composition during aging which was sufficiently rich in Mn to enter a range of lattice instability upon cooling to room temperature.

C. STATEMENT OF THE PROBLEM AND HYPOTHESES

The central problem is that in spite of the mass of research that has been conducted on the Cu-Mn alloy system, the exact mechanisms that cause high damping and/or tweed contrast in aged, low Mn based, Cu-Mn alloys are unknown. As a result, no one knows how to optimize properties by alloying and heat treatment for the development and maintenance of high damping.

Although the exact reasons for the known effects of time, cycling, deformation and temperature have not been determined, there are several working hypotheses which can be stated as a starting point to address the problem.

1. Hypothesis One

The tweed contrast microstructure that is observed in certain aged Cu-Mn alloys is due to either:

- a. Distortion caused by the creation of a dispersion of Mn-enriched regions in a Cu-rich matrix; or
- b. Distortion due to the FCC-to-FCT transformation occurring within the Mn-enriched regions when they are cooled below the local Neel temperature.

Question: Which, if either, of these mechanisms is operative?

2. Hypothesis Two

Based on the fact that quite high damping can be developed in aged Cu-Mn alloys, the mechanism of high damping in aged Cu-Mn alloys must be associated with one or more of the following:

- a. Stress-induced transformation and twinning (FCC-to-FCT).
- b. Stress-induced movement of the FCT:FCT twin boundaries.
- c. Some sort of subtle fine-scale response within a tweed microstructure.

Question: Which mechanisms provide high damping in this alloy or can be eliminated from hypothesis two?

3. Hypothesis Three

In any case, damping is a function of prior heat treatment (i.e., aging time and temperature). In general terms, this must be because either:

- a. Some critical feature is developed in the microstructure; or
- b. The ability to operate a critical mechanism is optimized.

Question: Can detailed microstructural observations point out the key feature of the mechanism?

4. Hypothesis Four

In specific terms, creation of a "responsive" material must be linked in some way to optimization of the microstructure, and this must be keyed to the role of the Mn-enriched regions created during aging. These may play one of the following roles:

- a. To trigger the formation of FCT plates upon cooling after the aging treatment.
- b. To trigger the formation of FCT plates when the material is subjected to (cyclic) stresses.
- c. Or the Mn-enriched regions themselves, or regions nearby, absorb the vibrational energy locally by some unknown mechanism.

Question: Is there observable evidence which may help to sort out the role of the Mn-enriched regions in the damping mechanism?

5. Hypothesis Five

Because it is known from previous reports that high damping is not necessarily stable in aged Cu-Mn-based alloys, microstructural optimization by aging must be a delicate and subtle matter, and probably very fine scale.

Question: Will the apparent subtlety and fine scale of certain aspects of the microstructural mechanism allow visual evidence to be collected?

6. Hypothesis Six

In this particular high damping alloy, there seems to be a unique combination of conditioning requirements (for the high damping) which involve both a replacive phase transformation step (involving a critical combination of aging time and temperature) and a displacive step (involving the operative transformation temperature upon quenching). The aging step apparently has to do with balancing the competitive replacive phase transformations (i.e., equilibrium α -Mn phase formation, spinodal decomposition, and coarsening).

Question: How do the kinetics of the replacive phase transformations interact?

7. Hypothesis Seven

The effectiveness of the displacive step is keyed to:

- a. Prior heat treatment.
- b. The temperature to which the material is quenched.
- c. The percentage of the microstructure that is transformed from FCC-to-FCT.
- d. The ability of the Cu-rich regions to cooperatively respond to shear.

Question: Can visual evidence be obtained to confirm the displacive response?

D. OBJECTIVE

The purpose of this research was to use transmission electron microscopy to study the details of microstructural changes which occur upon aging the 53Cu-45Mn-2Al alloy and provide answers, insights or other information relevant to answering the questions posed by the above hypotheses.

II. EXPERIMENTAL PROCEDURES

The alloy examined was provided by Olin Metal Research Laboratories in the form of 25mm thick plate. The alloy composition was confirmed by Anamet Laboratories, Berkley, California to be:

	Cu	Mn	Al	Zn	Si	Fe	Cr	Remain
w/o	53.1	44.8	1.61	0.1	0.08	0.06	0.05	0.2

Discs of 0.5mm thickness were sliced from 3mm diameter rods of the material with a low-speed diamond wafering saw. The discs were then hand sanded to a thickness of about 0.25mm. These specimens were solution heat treated at 800 C for 2 hours in evacuated quartz tubes and water quenched. Subsequently, specimens were aged at 400 C for various times and water quenched once again. In order to prevent possible room temperature aging [Refs. 23,35], specimens were stored in a freezer at -22 C after each heat treatment stage, and were not removed until it was time to prepare TEM specimens.

Thin foils were prepared by a careful two stage process. Following aging, each disc was lightly sanded to remove any oxide layer that may have formed. The disc were then dimpled in a lollipop holder by jet electropolishing with a solution of 50% H_3PO_4 and 50% H_2O at room temperature and a current of about 580ma. Final thinning to perforation was accomplished by holding the specimen with platinum tipped tweezers and static electropolishing in a magnetically stirred solution of H_3PO_4 saturated with CrO_3 at 8v to 12v and 20 C to 30 C. All thin foils were observed immediately after electropolishing. Observation was carried out using a JEOL-120CX transmission electron microscopy operated at 120kv. Further information on the procedures and equipment set up used for preparation of thin foils is contained in Appendix A.

III. RESULTS AND DISCUSSION

For relatively short aging times (for example, less than 4 hours at 400 C), "mottled" contrast is observed in samples subsequently quenched to room temperature, as seen in Figure 3.1. The fact that tweed contrast is not displayed indicates that the conditions for tweed [Ref. 26] have not (yet) been satisfied. That is, there are either not enough points



Figure 3.1 Mottled contrast in sample aged hrs. at 400 C. Bright field image with $g=00\bar{2}$ Beam direction near $\langle 100 \rangle$.

of asymmetric strain dispersed in the microstructure, and/or the degree of asymmetric strain provided by each point is not sufficient. At an aging temperature of 400 C, the present alloy composition probably places the alloy inside the Cu-rich side of the miscibility gap. Aging at this temperature would therefore be expected to produce a dispersion of FCC Mn-enriched clusters in an FCC Cu-rich matrix. The development of such a dispersion may in itself

constitute the array of strain centers required to perturb the matrix into a tweed contrast, but if the clusters remained FCC, it is likely that the distortion would be isotropic, and aligned contrast traces, if they developed, would be $\{100\}$, not $\{110\}$. If, however, it is assumed that the potential source of asymmetric straining points is the distortion of the incipient FCC-to-FCT lattice transition occurring within these Mn-enriched regions (such a distortion would constitute the asymmetric straining points required for a $\{110\}$ tweed contrast), then it is apparent that at these early times the phase separation in the FCC solid solution has not yet produced sufficient numbers of Mn-enriched regions or that perhaps these regions have not yet achieved the degree of Mn enrichment required to undergo a tetragonal distortion upon cooling to room temperature.

Aligned $\{110\}$ tweed contrast was observed in all samples aged 4 hours or longer at 400 C. An example is presented in Figure 3.2. The image contrast has all the typical

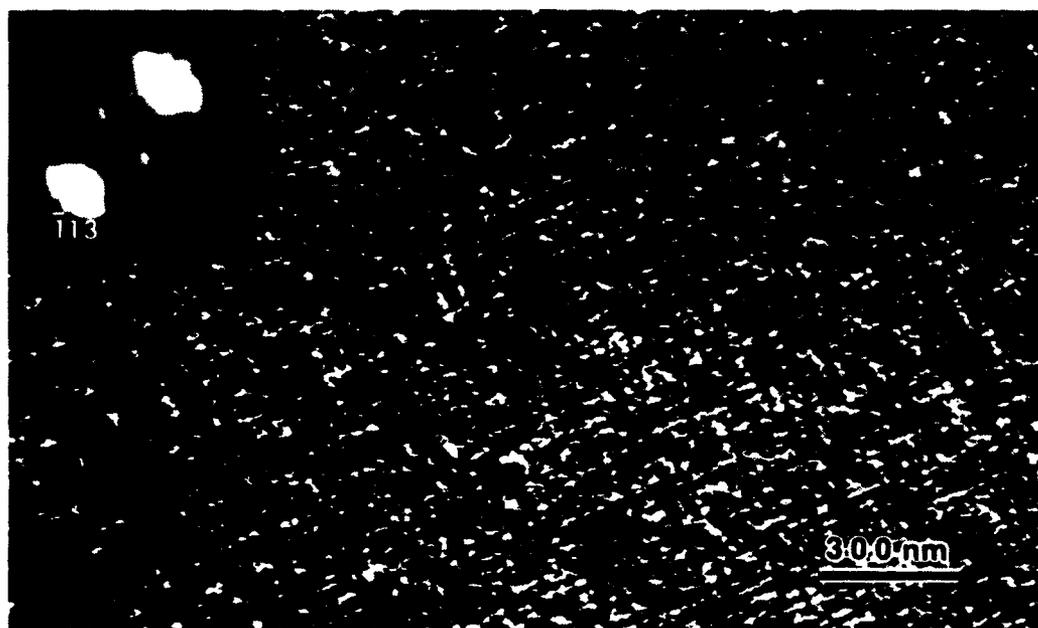


Figure 3.2 Tweed contrast on sample aged 4 hrs. at 400 C. Dark field image with $g=113$. Beam direction near $\langle 110 \rangle$.

characteristics of tweed which have been outlined by Tanner [Ref. 26] and by Robertson and Wayman [Ref. 27]. For example the expected image extinctions are observed: a given set of the near-parallel striations within the tweed becomes invisible if the operating g -vector is perpendicular to the trace direction. Also, the observed spacing of the striations is a sensitive function of foil orientation. Very slight tilting, while maintaining the same operating reflection in a two beam condition, can significantly change the apparent spacing of the striations. The observed spacing is also a function of the degree of deviation from the Bragg condition, as observation of the tweed near a bend contour reveals, and of the effective extinction distance. The effect on diffraction contrast of different g -vectors is shown in Figure 3.3; in these two images, the FCC annealing twin boundary may be used as a marker. While the analysis has not been as thorough as that of Robertson and Wayman,

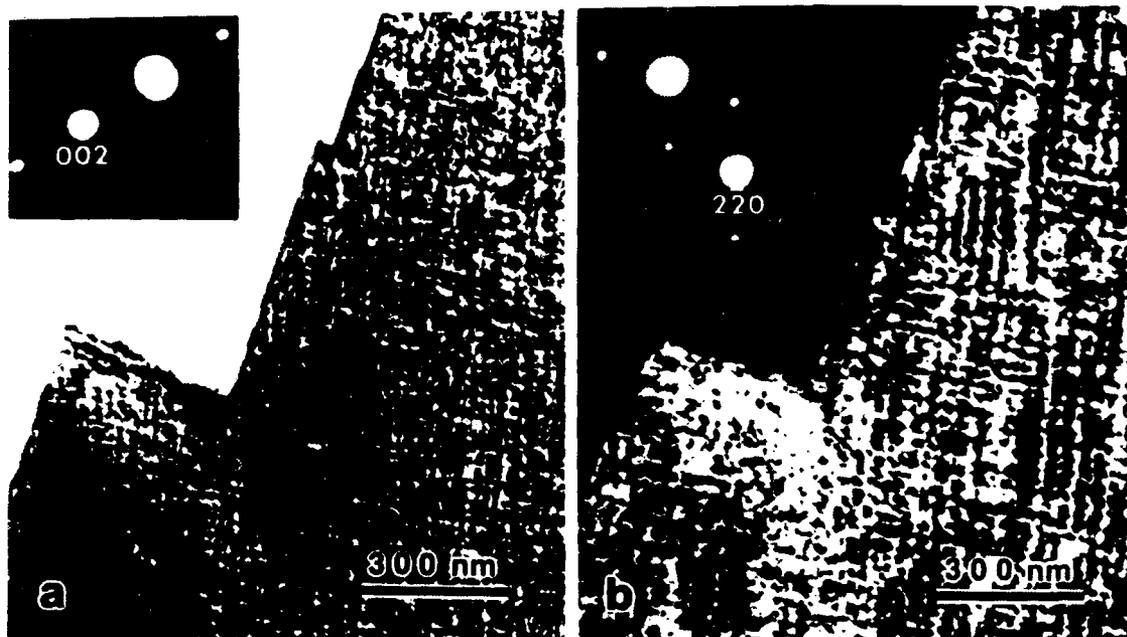


Figure 3.3 Area in sample aged 10 hrs. at 400 C, imaged in bright field with two different near two beam conditions; for both images the beam direction is near $\langle 110 \rangle$; (a) $g=002$, (b) $g=220$.

the observations would seem to agree in every respect with the "rules" which they outline for tweed contrast microstructures.

As aging was extended to longer times an enhancement of the aligned contrast was observed, taking the form of bands of darker contrast, as seen in Figure 3.4. This sort of contrast, which is not uniform over the thin foil sample, begins to be noticed for aging times of 8 hours or more at 400 C. This appears to be similar to observations made by Shimizu and coworkers [Ref. 34] during cooling of a Mn-rich alloy (Mn-26 atomic % Cu). The present observation indicates that even in the composition-segregated microstructure of the aged alloy, an increasing lattice instability is reflected by contrast which extends across both Mn-enriched and Mn-poor regions. These bands may be interpreted as regions in which the strain of the incipient FCC-to-FCT lattice transformation is enhanced, eventually to lead to

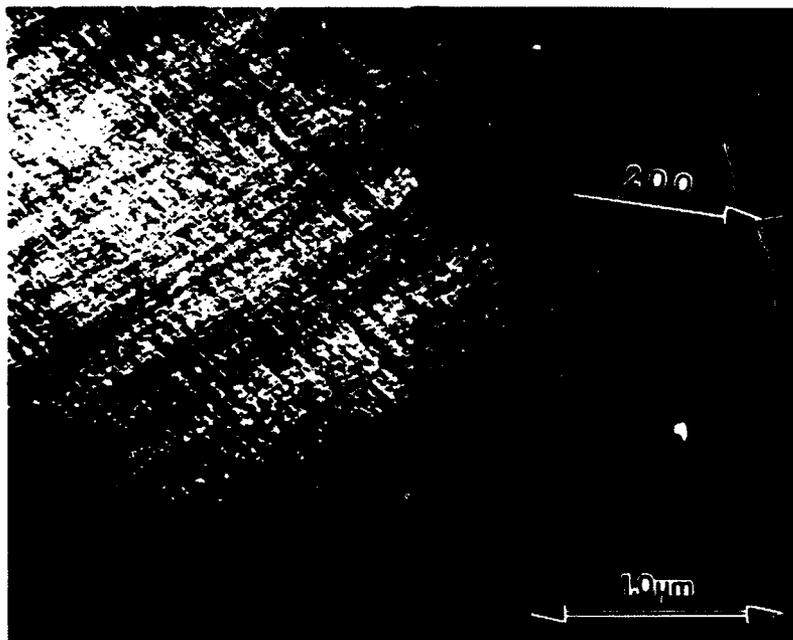


Figure 3.4 Banded tweed contrast in sample aged 8 hrs at 400 C. Bright field image. Beam direction near $\langle 110 \rangle$. (Photo by Dr. M. H. Wu)

the development of definite FCT lamellae [Ref. 34]. These "tweed bands" obey the same extinction rules as the set of tweed striations to which they are parallel, that is, they become invisible when the operating g -vector is perpendicular to the trace direction [Ref. 36]. This is consistent with the general rules for tweed contrast outlined by Robertson and Wayman [Ref. 27], and indicates that the source of the banded contrast is strain of the same sense as that which creates the general tweed contrast. This leads to the conclusion that the lattice instability is becoming more pronounced.

Another observation with increasing aging is a change from stress-induced slip to stress-induced lattice transformation. At early aging times, occasional stress concentration points in the form of notches at the edge of the thin foil perforation, were observed to induce slip bands in the FCC matrix phase, as seen in Figure 3.5a. This

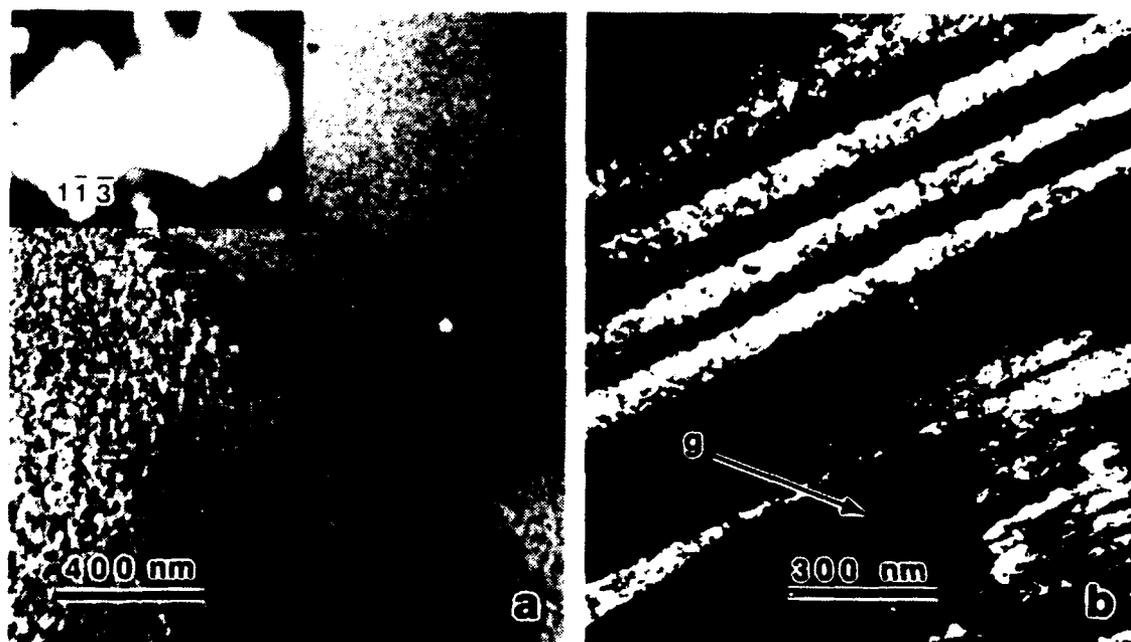


Figure 3.5 (a) Slip trace in sample aged 2hrs at 400 C. Bright field; $g=113$; Beam direction near $\langle 110 \rangle$
 (b) Twins in sample aged 16 hrs at 400 C. Dark field; $g=020$; Beam direction near $\langle 100 \rangle$.

indicates that the FCC phase at this point is relatively stable, not prone to stress-induced lattice transformation. At longer aging times, say 14 hrs, stacks of parallel twins begin to form, as seen in Figure 3.5b. The ability to produce these features also appears to reach a maximum as aging proceeds, for example around 16 to 22 hours for aging at 400 C, which happens to correspond to the condition for maximum damping capacity in this alloy [Ref. 3,36]. These static twins represent a further stage of lattice instability of the aged FCC matrix phase. Vintaykin and coworkers [Ref. 37] have quantified the critical shear stresses for slip and twinning in certain binary alloys. Their findings may be interpreted as indicating that the formation of the twinned FCC condition, that is the quasi-martensitic state, reflects a relative decrease in resistance to twinning relative to slip, which is consistent with our observations on the present aged alloy.

In the course of examination of the tweed microstructure over a range of aging conditions, a unique and remarkable observation was made, one which has not previously been reported in connection with Cu-Mn or any other tweedy alloy. It was noticed that certain tiny areas within the tweed contrast were not completely static. Rather, as the tweed was observed on the viewing screen at sufficient magnification (say 40,000X or so), certain specific points within it were observed to "flicker". This effect was not observed in samples at early aging times, when the structure displayed simply a "mottled" contrast, but only after the aligned tweed traces became defined.

The "flickering" effect consists of spatially consistent and repetitive contrast variations, that is, only certain points flicker, and the contrast variation is always very much the same in nature (although the frequency is not constant). The regions that flicker are on the scale of the tweed spacing, around 10 to 20 nm. Upon close examination,

it is apparent that the contrast variations are not of an "on-off" nature, but rather involve short movements or rotations of lines of contrast on about this scale, as seen in Figure 3.6. As there are a great many of these regions active on the viewing screen at any one time, it is not easy to make a complete quantitative characterization of the activity. It also presents certain problems in photographic recording by the usual timed exposure methods, but videotape recordings have been made. The most successful method for isolating the sites and the character of the contrast shifts has been the use of weak beam dark field imaging techniques, as exemplified by Figure 3.6a, where a very specific geometric form can be seen, which includes a distinct V-shaped feature.

It must be emphasized that the dynamic nature of the flickering image has quite a different character than the phenomenon which has been termed "shimmering" in various

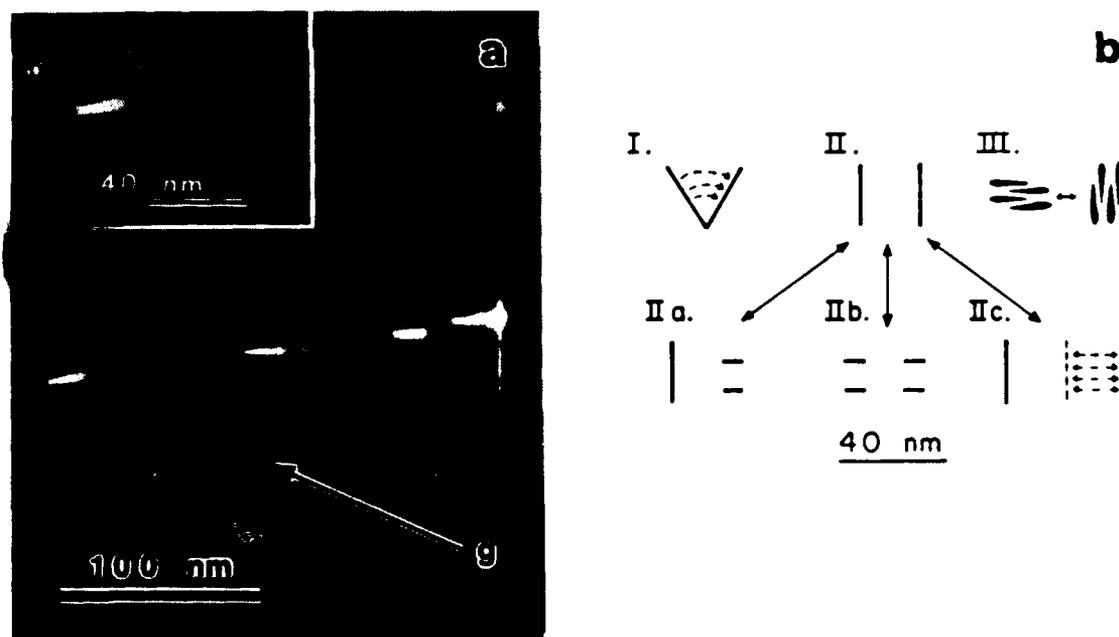


Figure 3.6 (a) Flickering regions, aged 10 hrs. at 400 C. Weak Beam; $g=222$. Beam direction near $\langle 110 \rangle$.
 (b) Schematic of a variety of flickering morphologies which are observed.

alloy systems [Refs. 38,39,40]. A detailed model for the origin of this unusual effect has not yet been developed, but it is apparent that the underlying cause of the contrast variation is a subtle degree of lattice distortion in quite small regions of the microstructure, these presumably being the Mn-enriched regions resulting from the phase separation. The initial impression is that the flickering contrast is a manifestation of quite small sections of crystal, probably less than 10nm in size, flipping a tetragonal distortion from one c-axis orientation to another. This would be consistent with the Mn-enriched regions having entered a pre-transformation range wherein the lattice begins to mimic the incipient FCC-to-FCT quasi-martensitic transformation. This is a notion which is in accordance with recent ideas, such as presented by Barsch [Ref. 41], Krumhansl [Ref. 42], Tanner [Ref. 43] and others, regarding the very common development of incommensurate lattice structures in a "pre-martensitic" temperature range, creating lattice distortions which effectively anticipate the martensitic transformation which may follow [Ref. 30].

The only previous report found of a dynamic TEM image contrast effect similar to the present observations was in connection with the creation and annihilation of omega domains in a Ti-Mo alloy [Ref. 44]. For Cu-Mn alloys, evidence does exist that the phonon spectra is appropriate for the display of pre-martensitic effects of this kind [Ref. 45]. It is therefore proposed that the flickering contrast effect reported here is a manifestation of the incipient FCC-to-FCT lattice transition which occurs in quite small regions of appropriate composition in the aged microstructure. It would also seem that in view of the present observations, it may be unreasonable to rule out the possibility that in certain pre-martensitic alloys the phenomenon of "shimmering" is simply a denser concentration of the uniquely localized (because of the composition

variations in the parent phase) distortional events that here are termed "flickering".

The flickering activity varies systematically with aging, and there are a number of indications that coupling can occur between neighboring regions of distortion, thus progressing another step along the path toward FCT martensite. One of these observations is that at relatively short aging times (4 to 8 hours at 400 C) the localized flickers often erupt into linear "flashes" along one of the (110) traces in the tweed, with a length of perhaps 10 to 20 tweed spacings (some 100's of nm). These miniature plate-like features repeatedly form and revert. If it is assumed that this sort of event originates in one or more Mn-enriched regions, then it is apparent that the lattice transition, once it has initiated, is able to propagate across a distance which must be longer than the wavelength of the composition modulation. This may be possible if at early times the dispersion of Mn-enriched regions consist of small but quite closely spaced regions, allowing a coupling of the distortions which develop in each of them. This is consistent with x-ray diffraction result reported by Vintaykin and coworkers [Ref. 46] for aged Cu-Mn-Ge alloys, which indicated that there is a modulation of the FCT c-parameter corresponding to a composition modulation in the parent phase.

It is also noticed that at longer aging time (greater than about 8 hours), when the dispersion of the Mn-enriched regions is known by neutron studies to coarsen rapidly [Ref. 47], flicker sites become confined to a size of about 20 to 40nm; the linear "flashes" into and out of the small (110) plates are not seen at all. This suggests that the lattice distortion of a given region is not able to trigger a neighboring region, at least not on an obviously cooperative manner, unless the unstable regions are close enough together.

In the present study, a static indication of cooperative interaction between the distortional centers which underlie the tweed contrast was previously discussed. This is the aligned contrast taking the form of bands of darker contrast seen in Figure 3.4. It therefore seems that the general sequence of observed morphologies as the lattice instability becomes increasingly manifested is: "mottled", "tweed", "tweed bands", and finally FCT twins. The flickering activity apparently reaches a peak during the tweedy stages.

A complicating factor in the aging of these alloys is that phase separation in the FCC solid solution within the miscibility gap is inherently in kinetic competition with the long-range diffusional reaction to form the equilibrium α -Mn phase. This essentially pure Mn phase was observed to form as particles which are quite large relative to the tweed spacing. When this occurs, the α -Mn particles may be expected to naturally drain off Mn from the rest of the microstructure, leading to a decrease in the amplitude of any fluctuation in Mn composition. This would then in turn be reflected in a lesser degree of lattice instability, that is, a lesser tendency for the FCC-to-FCT transition, as well as a decrease in the demonstration of any possible precursor events to it, such as flickering.

At an aging temperature of 400 C, the 53Cu-45Mn-2Al alloy was indeed in the process of forming observable amounts of α -Mn over the very range of aging time (8 to 24 hours) where a tweed contrast microstructure was becoming more defined, a distinct tendency to form stress-induced twins was being demonstrated, and the damping capacity was rising to a maximum value. Therefore, an attempt was made to produce an aged condition in which the microstructure demonstrates a tweed contrast, but contains few, if any, α -Mn particles. This was achieved, for example, for samples aged 10 hours at 450 C, and it is revealing to compare the microstructure of this condition, shown in Figures 3.7 and

3.8, as well as the flickering activity, with microstructures in which α -Mn particles are present and apparently acting as Mn sinks.

One feature of the tweed microstructure seen in Figure 3.7 which is different than, say, that of Figure 3.2, is a less defined pattern of $\{110\}$ tweed traces. Close examination of Figure 3.8, reveals that is because the features underlying (and causing) the tweed contrast are somewhat larger and not so finely dispersed. The most vivid features seen in Figure 3.8 are the small sets of zig-zag patterns. These are localized, presumably to the most Mn-enriched regions, and the most distinct groups of this sort were not observed to be flickering. It is therefore assumed that they represent static groups of self-accommodating FCT units. They are very reminiscent in form, but much smaller in size, to the type of self-accommodating martensite plate groups which are well-known to form for many thermoelastic martensites [Ref. 48]. The V-shapes within these groups seem to be pointing along one or the other of the two obvious $\{110\}$ tweed traces in this $\langle 100 \rangle$ beam direction image. The obvious indication is that the habit plane traces of the V-shaped crystals in these zig-zag groups are clustered fairly closely around one of the $\{110\}$ parent phase planes, and in fact seem to be symmetrically disposed with respect to these parent planes.

Surrounding these zig-zag groups in the tweed contrast microstructure were numerous strongly flickering regions. The contrast variation in a given region exhibited a consistent pattern which often consisted of abrupt rotations of approximately 90 degrees of a small zig-zag group or a short line (or pair of lines) of contrast. For example, the V-points in a zig-zag group, or the alignment of the short lines of contrast, would suddenly switch back and forth from pointing along one or the other of the two orthogonal $\{110\}$

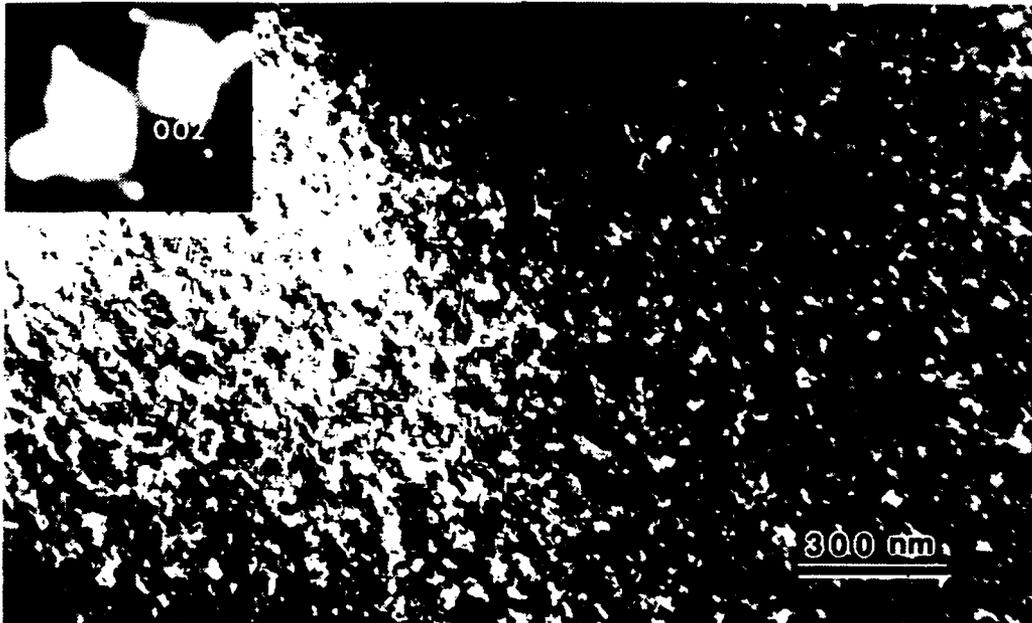


Figure 3.7 Tweed microstructure in sample aged 10 hours at 450 C. Bright field image. Beam direction near $\langle 100 \rangle$.

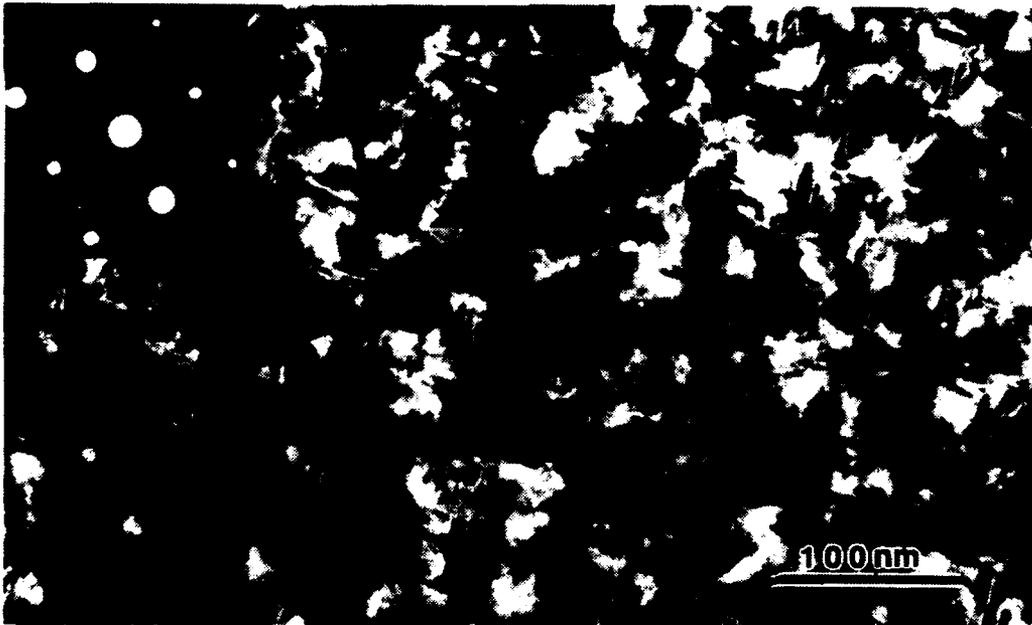


Figure 3.8 Static and flickering zig-zag groups in a sample aged 10 hrs. at 450 C. Beam direction near $\langle 100 \rangle$.

tweed traces. The static zig-zag groups and the strength and distinction of the flickering contrast were not seen in some other quite well-defined tweed microstructures. It is concluded that this is due to the relative effects of the competitive long-range diffusional nucleation and growth kinetics of the formation of the equilibrium α -Mn phase.

Supporting evidence that the zig-zag groups are FCT units can be obtained from the selected area diffraction pattern (SADP) presented in Figure 3.9a. Splitting of the 002 and 022 diffraction spots indicate the presence of the FCT phase. Although this SADP was taken from alloy aged for 10 hours at 450 C, Figure 3.9b shows evidence that the FCT phase also exist in the alloy aged at 400 C, where these same diffraction spot have elongated but not split. This is consistent with the fact that the underlying microstructure is not as well defined. The Cu-Mn alloy system is analogous to the Fe-Pd, Fe-Pt and In-rich alloy systems [Refs. 49,50,51] and it has been shown by previous workers

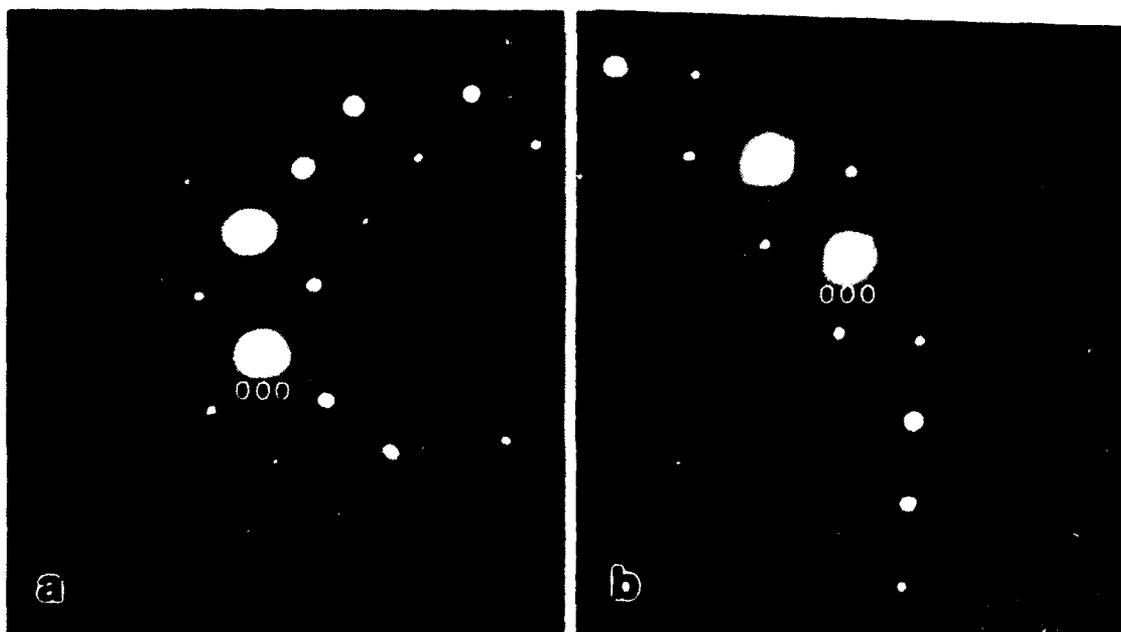


Figure 3.9 (a) SADP from sample aged 10 hrs. at 450 C.
(b) SADP from sample aged 12 hrs. at 400 C.

[Refs. 12,21,23] that the lattice parameter varies as a function of temperature. That is, the degree of tetragonality increases gradually with decreasing temperature at a given composition. In the present alloy the composition most likely varies from Mn-enriched region to Mn-enriched region, although there is an average composition and a limiting composition which all regions are trying to reach. Since the Neel temperature and FCC-to-FCT transition temperature are therefore different in each region, it is logical to assume that there is also a variation of tetragonality within the regions, hence the elongated diffraction spot. As more and more Mn-enriched regions reach the limiting composition, then the tetragonality of the structure approaches a limiting value and the diffraction spot splits.

The comparison between the microstructures aged at 400 C and 450 C leads to the proposal that the formation of the equilibrium phase α -Mn is in some way associated with the decrease in damping which occurs upon extended aging at temperatures within the miscibility gap. Two separate processes are believed to contribute to this effect: (1) the draining of Mn from the Mn-enriched regions as the α -Mn forms and, (2) interference with the formation and propagation of the FCT microstructure. It is not clear at this point whether the α -Mn forms within the Mn-enriched regions or on the boundaries of these regions, or at both types of locations.

IV. CONCLUSIONS

It has been shown that tweed contrast microstructure is observed after the alloy is aged for about 4 hours at 400 C. It is most likely that this contrast is due to the FCC-to-FCT transformation occurring within the Mn-enriched regions as indicated by a change from stress-induced slip to stress-induced transformation, evidence of FCT microstructure from the selected area diffraction patterns and the flickering phenomenon.

Damping is most likely caused by stress-induced transformation and twinning and stress-induced movement of twin boundaries. It is also possible that damping is associated with the flickering phenomenon, as the lattice cycles back and forth between different c-axes. Although these mechanisms have been observed, there is insufficient data to positively rank their relative importance. The impression is that the stress-induced phase transformation, which seems to be most readily accomplished around 14-16 hours aging time, is most likely of prime importance. Since a relatively large increase in damping is achieved at just about these aging times, other mechanisms may be of lesser importance.

The flickering phenomenon appears to be a prime example of a premartensitic effect where the parent phase (in this case FCC Mn-enriched regions) prepares itself by developing periodic, incommensurate displacement patterns that mimic the new product phase structure (in this case twinned FCT).

Finally, based upon the preceding discussion, the following aging sequence for this alloy is proposed:

- a. Nucleation of Mn-enriched regions occurs very quickly (approximately within the first 10 minutes of aging time) [Ref. 47].

- b. For times less than about 4 hours, the Mn-enriched regions grow in size. During this period, there are either not enough regions to produce the strain necessary for tweed microstructure or the regions have not reached sufficient Mn-enrichment to support transition to FCT.
- c. For times greater than about 4 hours but less than about 8 hours, some of the Mn-enriched regions are in a premartensitic condition and form an unstable FCT. The spacing between regions is still relatively close, such that a Mn-enriched region can "trigger" a cooperative transformation with several other Mn-enriched regions.
- d. For times of about 8 hours or longer, a coarsening process has reduced the number of Mn-enriched regions and increased their average separation distance such that an obvious cooperative transformation can no longer take place.
- e. For times of about 14 hours or longer, some of the Mn-enriched regions have attained a Mn-enrichment such that the localized Neel temperature is above room temperature and these regions can form a stable FCT phase. The alloy is prone to stress-induced transformation for aging times from 14 to about 22 hours: these are the aging times where the highest damping occurs.
- f. Beyond 22 hours, the damping decreases. This appears to be the point where formation of the equilibrium α -Mn begins to drain Mn from the Mn-enriched regions and possibly interfere physically with the damping mechanisms.

V. RECOMMENDATIONS FOR FURTHER STUDY

The following recommendations for further research are provided:

- a. The flickering phenomenon needs further investigation. Research using a heating and cooling stage on the TEM will aid in further characterizing this feature. This phenomenon appears to provide a rare opportunity to look further into premartensitic effects and in particular movement of the lattice. This research may require the use of HREM.
- b. Further studies involving other heat treatments would be beneficial. For example, a study in which the alloy is aged for 10 hours and the temperature is varied by 10 C to 20 C for each heat treatment over a range from 350 C to 600 C could be conducted and damping could be correlated with the microstructural changes.
- c. Additional investigation of room temperature aging is required. All previous research has been done on beams that have been optimally aged to produce peak damping. Question: what are the characteristics of material that has been, for example, underaged, but that still provides usable damping (i.e., 20-30%)?
- d. Studies on the effects of further alloying (e.g., Ga, Ge) on damping and room temperature aging should be conducted.
- e. An investigation into the corrosion characteristics and methods of protection of the alloy should be conducted. Question: can this alloy be cathodically protected without adverse effects to other components within a system?

APPENDIX A

ELECTROPOLISHING 53CU45MN2AL ALLOY

The highly different electrochemical potentials for Cu, Mn and Al (-0.521v, +1.18v and +1.66v respectively) make electropolishing of this alloy difficult at best. The aluminum has been specifically added to the alloy to produce a corrosion inhibiting surface layer. The difficulty in electropolishing increases with alloy aging time as a result of forming the Cu-rich and Mn-rich areas as previously discussed and of precipitation of α -Mn particles. Preferential etching at grain boundaries and particles is a problem with the electrolyte used, but does not prevent getting good thin foils for aging times less than 28 hours. Thin foils were not obtained for samples aged 64 hours.

Many variables influence the results in electropolishing, including the following [Ref. 52: p. 119]:

- * Surface area of the specimen.
- * Orientation of sample and cathode.
- * Choice of cathode material.
- * Anode to cathode spacing.
- * Electrolyte age and temperature.
- * Flow or stirring rate.
- * Current density and voltage.
- * Time.
- * Method of removing the specimen from electrolyte.
- * Washing procedures.

All of these variables must be closely controlled and logged for future reproducibility. Experience has shown that even under the best circumstances, the success rate for good thin foil production on the aged alloy may be less than 30%.

Samples were initially jet polished by the double jet technique using a Tenupol-2. Struers Metalog [Ref. 53]

provides a good discussion of this technique. Electropolishing was accomplished immediately following jet polishing. The equipment set up was as shown in Figure A.1.

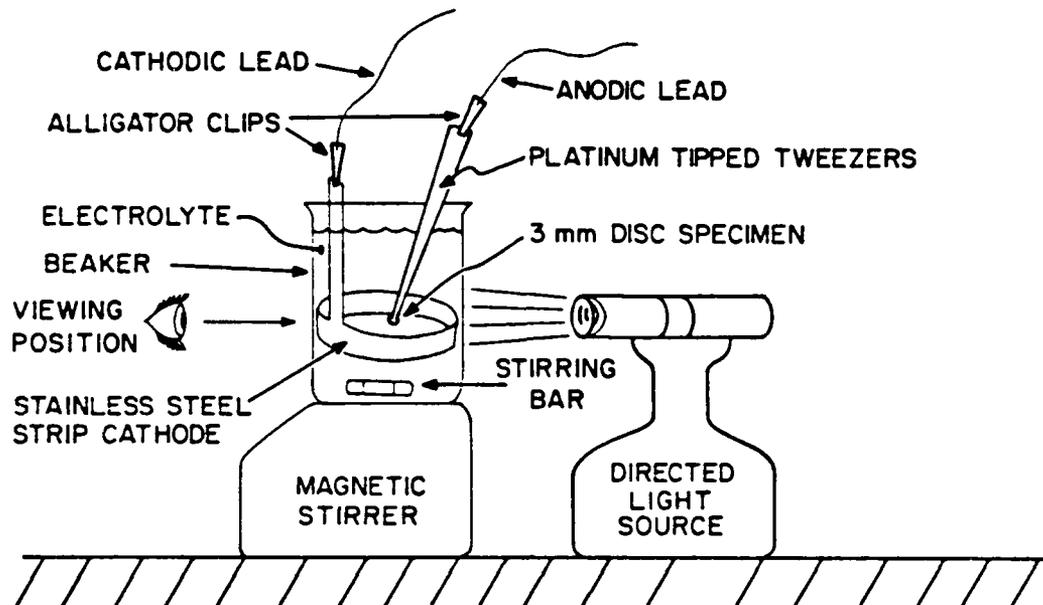


Figure A.1 Equipment setup for electropolishing.

The electrolyte solution was prepared by heating phosphoric acid to 40 C and adding CrO_3 while stirring continuously. CrO_3 particles were left in the bottom of the mixture even after cooling to maintain saturation. This solution was usually prepared several days in advance of polishing.

The key to this procedure is in using tweezers (Pt-tipped or stainless steel) to hold the specimen. The combination of alloy, electrolyte and stir rate lead to the generation of many bubbles and a lollipop holder is ineffective under these conditions.

The detailed procedures used were as follows:

1. Set up the equipment as shown in Figure A.1. The stainless steel cathode is placed near the bottom of the beaker with a tab above the beaker edge to connect as cathode.
2. Fill the beaker about two thirds full of electrolyte.
3. Fill a second beaker with distilled water for washing the TEM specimen after perforation.
4. Fill two petrie dishes with acetone and one petrie dish with methanol for the final rinse. Upon perforation, wash the TEM specimens as follows:
 - a. Upon removal from the electrolyte, first wash the specimen in distilled water.
 - b. Place the specimen in the first acetone bath for one minute.
 - c. Next, place the specimen in the second acetone bath for one minute.
 - d. Finally, rinse the specimen in methanol for one minute, and place on blotter paper to dry.
5. Using the tweezers, grip the specimen on an edge not jet polished and cinch with a plastic grip. Clip the anode lead to the top of the tweezers. Although the voltages used are low, rubber gloves should be used and all other electrical safety precautions followed.
6. Turn on the magnetic stirrer. A setting of 7 to 8 was used on a scale of 10.
7. Turn on the light so that it can be seen at some point to the side of the whirlpool and above the cathode.
8. Turn on the power supply and adjust the voltage as necessary. See Table A.1.
9. Place the tweezers in the solution with the TEM specimen in the light path and out of the whirlpool. Too high a stirrer setting creates too many bubbles and too low a setting causes too long a time before the specimen is perforated. Do not touch cathode with the specimen or the tweezers.
10. Pull the specimen out of the solution as soon as any light is seen through the specimen. Wash the specimen in the distilled water, disconnect the anode lead and complete the rinses. Place on the blotter paper to dry.

Adjustment of time, temperature, voltage/current or any of the other variables may be necessary to achieve good results.

TABLE A.1

CONDITIONS USED FOR STATIC ELECTROPOLISH

<u>Aged Condition</u>	<u>Voltage (v)</u>	<u>Temperature (C)</u>
Solution Treated	7, 8, 9, 10	23
400 C 1 hr	7, 8, 9	22
2 hr	8, 9, 10	24
3 hr	7.5, 8	24
4 hr	7.2, 8, 9	24
6 hr	9, 11	23
8 hr	8, 9, 10, 12	23
10 hr	11, 12	24
12 hr	12, 12+	24
14 hr	11, 12	20
16 hr	10, 10.5 10, 10.5	30 25
18 hr	10, 11.5	23
20 hr	9, 10	30
22 hr	10, 10.5	25
28 hr	11, 12	24
32 hr	9, 10.5	25
450 C 10 hr	11, 12	26
350 C 10 hr	10, 11	30

APPENDIX B

ADDITIONAL EFFECTS OF INTEREST IN 53CU45MN2AL ALLOY

The following features were seen but have not been studied in detail. They are provided here as an aid to follow-on researchers.

- (1) An interesting feature seen in the TEM photographs is the formation of both straight and wavy string-like contrast images as shown in Figure B.1a. Enlargement of the photograph, as shown in Figure B.1b, indicates an image that seems to be composed of plate-like features. The cause of this contrast is unknown and is worthy of further investigation.

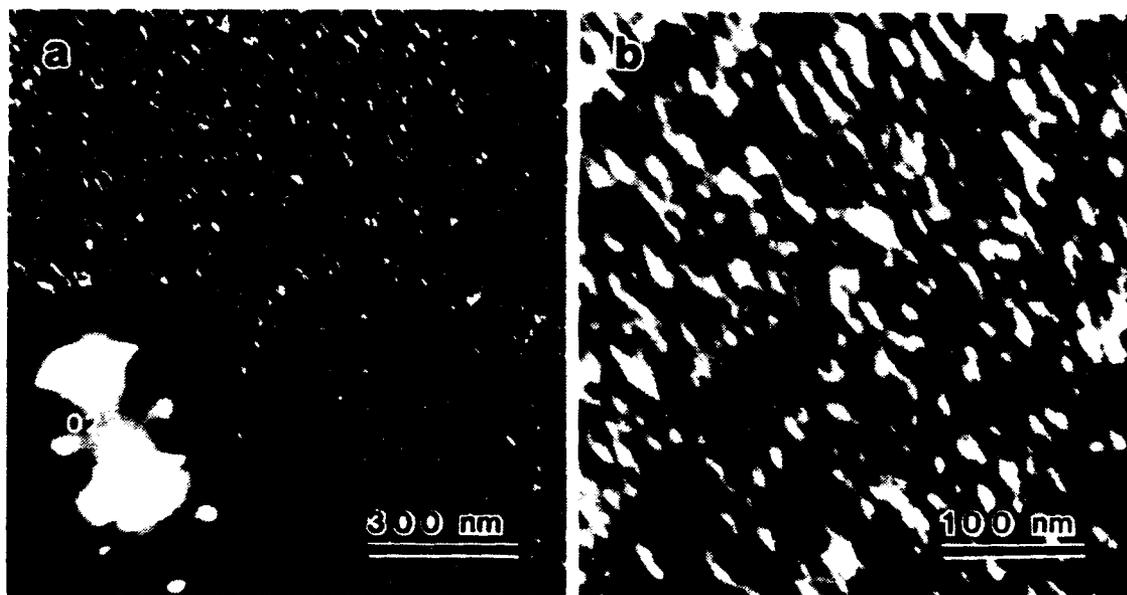


Figure B.1 String-like contrast in sample aged at 400 C for 4 hrs. (a) 68K (b) 194K

- (2) Some SADP's have satellites and a ring pattern that indicate the presence of an FCC structure with a lattice parameter of 4.4Å to 4.5Å. This is most likely MnO, which has a lattice parameter of 4.445 [Ref. 54].

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