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Abstract

The mechanical properties of two commercial high-strength, high-conductivity copper alloys have been investigated over a wide range of thermo-mechanical conditions. Vickers microhardness and miniature tensile specimen measurements were made on AMZIRC and AMAX-MZC in the cold-worked plus aged and annealed conditions. It was determined that a large portion of the strength of these alloys is due to their cold-worked nature, and this strength is lost when recrystallization occurs. The recrystallization temperature of both alloys is about 475°C for a 1 hour anneal, and is estimated to be about 320°C for a 20 year anneal. A linear correlation between microhardness and yield strength was observed for AMZIRC and AMAX-MZC, namely σ_y (MPa) ≃ 3.0 VHN.

Introduction

One of the key material parameters that is used to characterize potential fusion reactor materials is the yield strength. Unfortunately, there are many instances where it is impractical to irradiate conventional tensile specimens to obtain this information. The irradiation volume is often of limited size, and it is therefore desirable to be able to extract mechanical property information from nonstandard, subsized specimens [1]. One procedure for estimating the yield strength of irradiated metals is to make use of available correlations between microhardness measurements and tensile data [2]. The use of nondestructive microhardness testing also allows the irradiated specimen to be subsequently analyzed using other experimental techniques, e.g. transmission electron microscopy (TEM) [3].

Recent design studies have called for the use of high-strength, high-conductivity copper alloys in fusion reactors [4,5]. There are presently no known microhardness - yield strength correlations for these types of alloys. This investigation is intended to establish a working correlation between strength and hardness that should be applicable to most nonirradiated high-strength, high-conductivity copper alloys.

Experimental Procedure

Two commercial high-strength, high-conductivity copper alloys were selected for an investigation of their mechanical properties. AMZIRC (Cu-0.15% Zr) and AMAX-MZC (Cu-0.04% Mg-0.15% Zr-0.8% Cr) are heat treatable materials that have yield strengths of 400-500 MPa and electrical conductivities of 80-90% IACS (International Annealed Copper Standard) [6]. Foils of 250 μm thickness were obtained from AMAX Copper, Inc. in both the cold-worked plus aged (CWA) condition and in the solution annealed plus aged (SAA) condition.

The CWA heat treatment consisted of a solution anneal, followed by 90% cold-rolling and then aging for 30 minutes at 375°C for the AMZIRC alloy and 400°C for the MZC alloy. Some of these foils were subsequently solution annealed at 950°C for 100 hours and quenched in water (SA condition). The SAA heat treatment consisted of a solution anneal at 930°C for 45 minutes followed by aging for one hour at 450°C for AMZIRC and 500°C for MZC.

Specimens with dimensions of 0.5 by 5 cm were cut from the CWA foils of both alloys, mechanically polished, and then annealed for times ranging from 0.25-100 hours in a high vacuum furnace. Post-anneal cooling used a combination of furnace and air cooling. The specimen temperature (as monitored by chromel-alumel thermocouples attached to the specimen container) decreased by at least 100°C within one minute following the anneal. Different specimens of each alloy were used for each different annealing condition. The pressure in the vacuum chamber ranged from 1×10^{-6} to 1×10^{-9} torr during the anneal. All specimens were electropolished upon removal from the furnace in order to remove the oxide layer present from the anneal.

Room temperature Vickers microhardness measurements were made on the as-received and annealed specimens of both alloys at an indenter load of 200 g using a vibration-isolated Buehler Micromet microhardness tester. A minimum of nine different indentations in three widely separated areas were measured for each specimen. Selected specimens were examined in a JEOL TEMSCAN-200CX electron microscope. TEM disks were punched from the annealed foils and jet-electropolished using a solution of 33% HNO_3/67% CH_3OH cooled to -20°C at an applied potential of 15-20 V.

Miniature tensile specimens were punched from the as-received and annealed foils and deburred using procedures that are described in detail elsewhere [7]. The nominal dimensions of the gage section were 5.1 by 1.0 by 0.25 mm. The actual cross-sectional area for each tensile specimen was determined by measuring the width and thickness of the gage section at five different locations and averaging the results. A minimum of two tensile specimens were tested in the longitudinal direction for each of the annealing conditions in this study using a precision horizontal test frame with a free-running crosshead speed of 2.5 μm/s. Data for each tensile test were collected at room temperature in both a digital and analog manner. The 0.2 percent offset yield strength (σ_y) and ultimate tensile strength (UTS) were calculated using a computer based digital data acquisition program. The strength parameters of selected specimens were also calculated by graphical techniques using the analog data. The results were in good agreement with the computer-generated values.

Results

Table 1 lists the measured room temperature properties of AMZIRC and AMAX-MZC in the SA, SAA and CWA condition. Within the scatter of the data, there was no difference in the mechanical properties as measured in the transverse and longitudinal directions. The AMZIRC alloy develops high strength only after cold work plus aging, and shows minimal precipitation hardening. AMAX-MZC exhibits appreciable precipitation hardening, but once again most of its strength in the CWA condition can be attributed to cold work effects.

The microhardness numbers of AMZIRC and MZC specimens which were initially in the CWA condition are shown in Figs. 1 and 2 as a function of anneal conditions. The arrows indicate the microhardness numbers for the alloys in the solution annealed condition. The recrystallization temperature (i.e., the temperature where the microhardness decreases rapidly) depends on the annealing time and is about 475°C for both AMZIRC and AMAX-MZC for a one hour anneal. It has been empirically established that the recrystallization rate of metals follows an Arrhenius relationship [8]. The annealing data may therefore be plotted as a single curve by making use of the Larson-Miller parameter [9], as shown in Fig. 3 for AMZIRC. A similar curve was obtained for AMAX-MZC. Table 2 summarizes the extrapolated time-dependent recrystallization temperatures obtained from this analysis. The curves predict a recrystallization temperature of 320°C for both AMZIRC and MZC for a 20 year anneal, which is the maximum design lifetime of a copper alloy device in a fusion reactor [4,5].

The microstructures of the cold-worked plus aged copper alloys in their as-received state and after thermal annealing for 1 hour at 450°C and 500°C are shown in Fig. 4. A high matrix dislocation density is present in the as-received alloys. Recovery processes have occurred following a 1 hour anneal at 450°C, which results in a lower observed dislocation density. Well-defined grains containing a low density of dislocations are visible following a 500°C anneal, indicating that recrystallization has taken place. The average grain size after the 500°C anneal is larger in AMZIRC compared to AMAX-MZC.

Comparison of the measured Vickers microhardness number and yield strength of cold worked plus aged AMZIRC and AMAX-MZC specimens in their as-received and annealed states leads to a linear relationship. The yield strength-microhardness correlation plots for AMZIRC and AMAX-MZC are given in Figs. 5 and 6. Data for the alloys in the SA and SAA conditions are also included in these plots. A direct, linear correlation between Vickers microhardness (VHN) and yield strength (σ_y) exists over the entire range of conditions investigated. The correlation equations that described the least squares fit to the data are, for AMZIRC

$$\sigma_y(\text{MPA}) = 3.03\ \text{VHN} - 38 \quad (1)$$

and for AMAX-MZC,

$$\sigma_y = 3.00\ \text{VHN} - 17\ . \quad (2)$$

The fact that the correlation plots have a small nonzero intercept is believed to indicate that the correlations are not applicable for very low strength alloys (< 100 MPa yield strength). However, it should be noted that this strength level is less than the solution annealed yield strengths of the alloys.

Discussion

The general form of the microhardness annealing curves for both alloys (Figs. 1, 2) is in good agreement with published results in the literature on Cu-Cr and Cu-Zr type alloys [10-16]. It is well established that these alloys exhibit a large decrease in strength upon recrystallization from the cold worked plus aged condition. The exact value of the recrystallization temperature appears to depend on the prior thermomechanical history of the alloys.

A review of the literature reveals that Cu-Zr alloys (such as AMZIRC) exhibit very little precipitation hardening for various aging conditions [15-18], in agreement with the present results. AMAX-MZC has a higher strength and microhardness value than AMZIRC for all of the thermomechanical states investigated except for the solution annealed case. This indicates that precipitation hardening is more important in AMAX-MZC as compared to AMZIRC.

The average grain size and minimum thickness to grain size ratio (t/d) are given in Table 3 for the tensile specimens examined in this study. The number of grains across the smallest dimension of the tensile specimen was greater than twenty for all specimens except for the solution annealed and solution annealed plus aged alloys. A common rule-of-thumb is that at least ten grains across the smallest cross-sectional dimension of a tensile specimen are required to obtain bulk behavior [7]. Researchers have found that there is no grain size effect on the measured yield or tensile strength for miniature tensile specimens of austenitic or ferritic steel as long as $t/d > 3\text{-}5$ [7,19]. Therefore, the miniature tensile specimen results should be representative of the bulk strength except possibly for AMZIRC in the SA and SAA conditions and MZC in the SA condition.

A slight peculiarity was noticed in the elongation data of AMZIRC and MZC in the SA and SAA conditions (Table 1). The measured elongation to fracture was greater for the aged alloys compared to the solution annealed condition, as was the strength of both alloys. Ductility generally decreases when strength increases, but there are many exceptions to this rule [20]. The observed increase in ductility is probably due to grain size effects. Ductility is known to increase with decreasing grain size [20], and the SAA alloys have a much smaller grain size than the SA alloys (Table 2) due to different solution annealing treatments.

One valid criticism of the experimental procedure followed in this investigation is that all tensile specimens were punched from foils after they had been annealed. It is uncertain whether edge deformation due to punching has a significant effect on the measured mechanical properties of annealed miniature tensile specimens.

It is surprising that the correlation plot of yield strength and Vickers microhardness number is linear over the entire range of possible thermomechanical conditions for AMZIRC and MZC. As evidenced in Fig. 4, the microstructures of the two alloys changed dramatically following the various annealing schedules. The slope of the correlation plot should depend on the work-hardening coefficient [21,22], which increased significantly after annealing of the cold-worked plus aged alloys. The slope of the correlation plot was the same for both the precipitation-hardenable alloy (AMAX-MZC) and the alloy which does not exhibit appreciable precipitation hardening effects (AMZIRC). The derived correlation for these high-strength, high-conductivity copper alloys, $\sigma_y \sim 3.0$ VHN, is identical to the relation found for the strength increase in copper and copper alloys due to radiation hardening [23]. Similar correlation results have been reported for nonirradiated copper and other metals [21,24].

Conclusions

The recrystallization rate of cold-worked plus aged AMZIRC and AMAX-MZC copper alloys following short term thermal annealing apparently obeys an Arrhenius relationship. The recrystallization temperature for a 1 hour anneal is about 475°C for both alloys. The estimated recrystallization temperature for AMZIRC and AMAX-MZC for a 20 year anneal is about 320°C in the absence of stress or irradiation effects.

A linear correlation between yield strength and Vickers microhardness exists over a wide range of thermomechanical conditions for AMZIRC and AMAX-MZC. The relationship for both alloys is given by σ_y(MPa) $\simeq 3.0$ VHN.

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Table 1. Measured Physical Properties of AMZIRC and AMAX-MZC at 22°C.

Alloy	Heat Treatment	Micro-hardness	Yield Strength (0.2% Offset)	Tensile Strength	Elongation in 5.1 mm	Electrical Conductivity
AMZIRC	SA	50 HV	110 MPa	150 MPa	21%	--
	SAA	51 HV	140 MPa	220 MPa	27%	76% IACS*
	CWA	146 HV	440 MPa	500 MPa	9%	75% IACS
MZC	SA	46 HV	90 MPa	120 MPa	14%	--
	SAA	78 HV	220 MPa	260 MPa	23%	54% IACS
	CWA	168 HV	490 MPa	540 MPa	9%	59% IACS

* IACS = International Annealed Copper Standard

SA = solution annealed; SAA: solution annealed, then aged; CWA: 90% cold-worked, then aged

Table 2. Predicted Recrystallization Temperature (T_R) of AMZIRC and AMAX-MZC.

Anneal Time	1 Month	1 Year	10 Years
T_R	380°C	350°C	330°C

Table 3. Grain size and minimum thickness to grain size ratio (t/d) for miniature tensile specimens.

	Solution Annealed		Solution Annealed Plus Aged		Cold-worked plus Aged: Annealed 1 hr, 600°C		Cold-worked plus Aged: No Anneal	
	AMZIRC	MZC	AMZIRC	MZC	AMZIRC	MZC	AMZIRC	MZC
Grain size (μm)	300	270	60	26	12	9	<1	<1
t/d	1	1	4	10	20	28	>250	>250

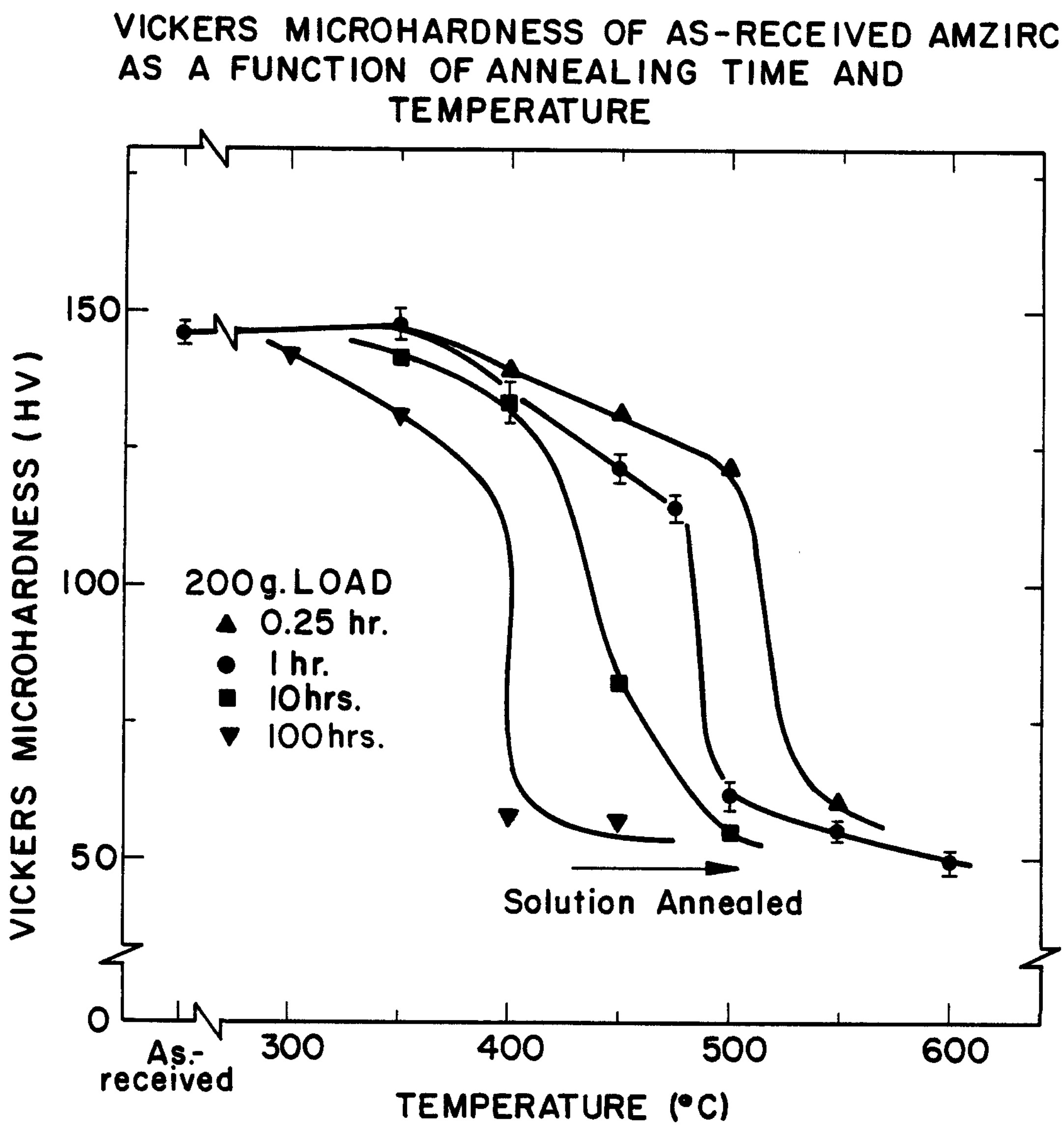


Fig. 1. Vickers microhardness of cold-worked plus aged (CWA) AMZIRC as a function of annealing time and temperature.

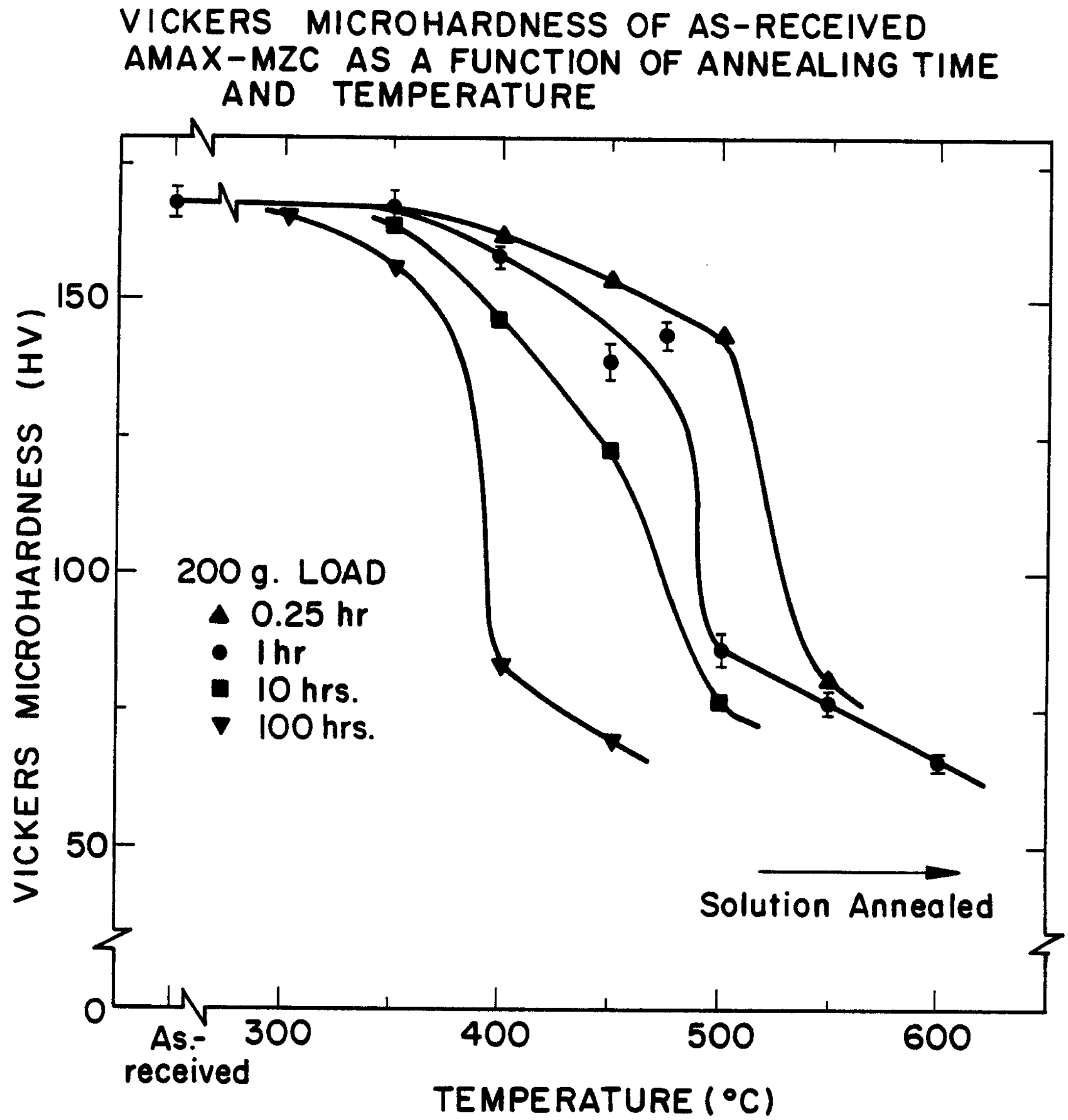


Fig. 2. Vickers microhardness of cold-worked plus aged (CWA) AMAX-MZC as a function of annealing time and temperature.

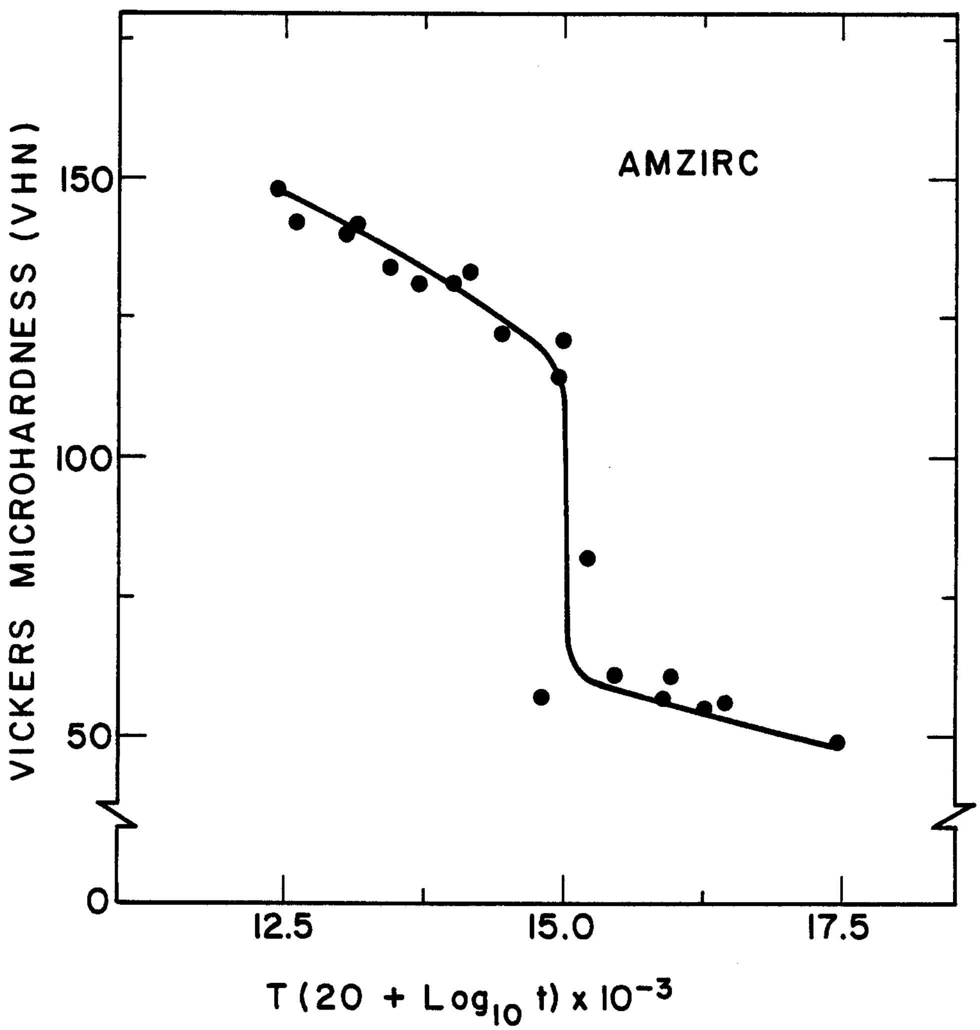
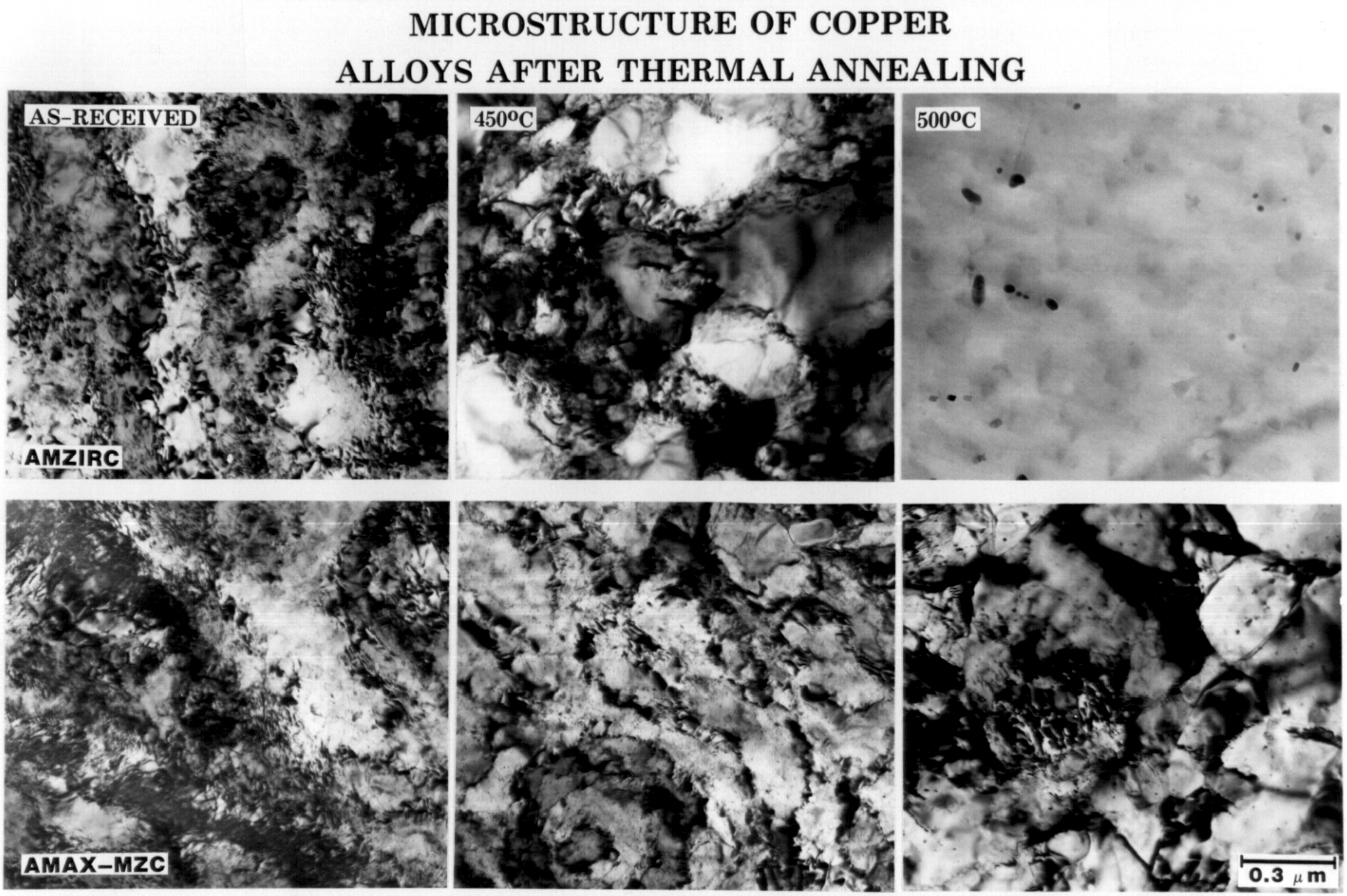


Fig. 3. Vickers microhardness of CWA AMZIRC plotted as a function of the Larson-Miller parameter. The curve contains all of the data shown in Figure 1.

Fig. 4. Microstructures of CWA copper alloys after annealing for 1 hour.



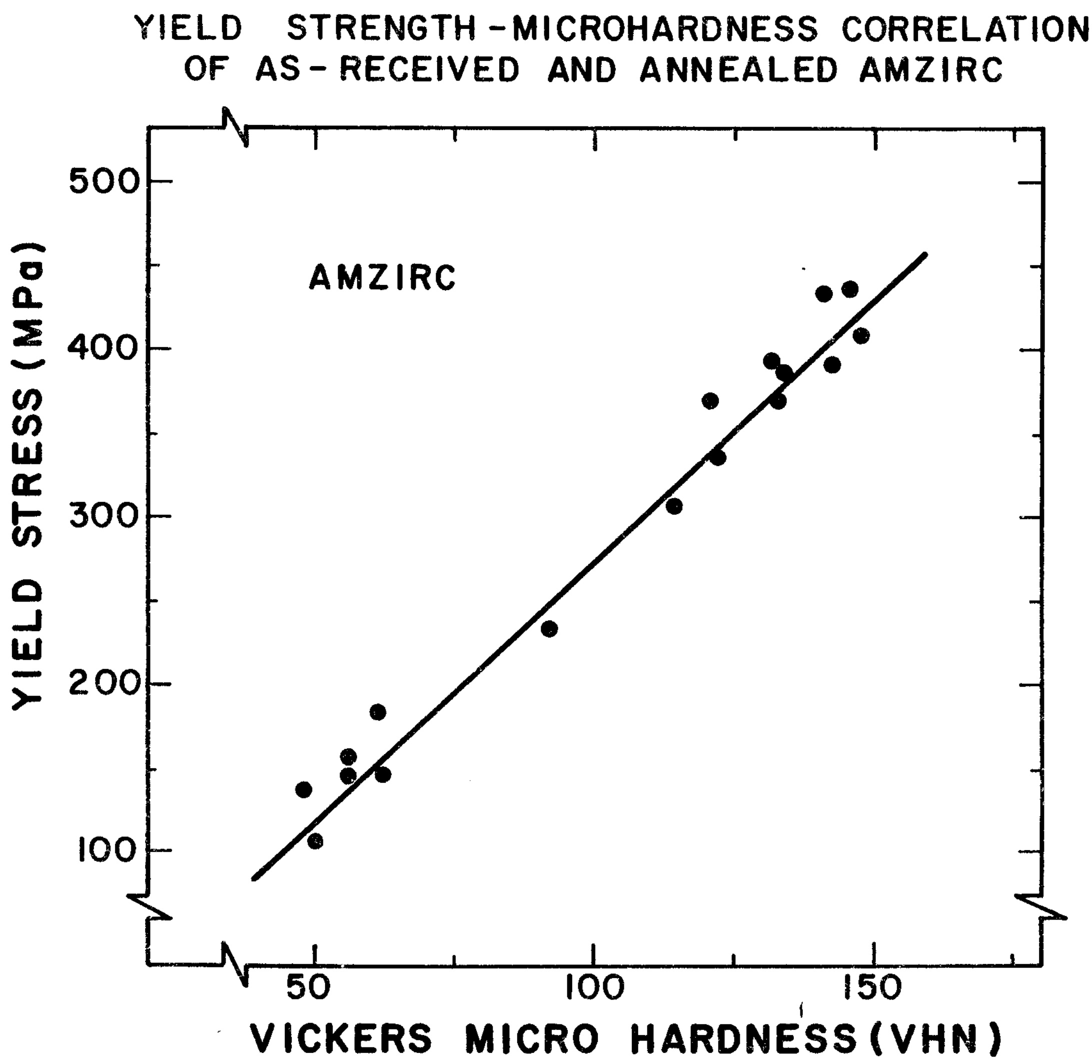


Fig. 5. Yield strength - microhardness correlation plot for CWA and annealed AMZIRC.

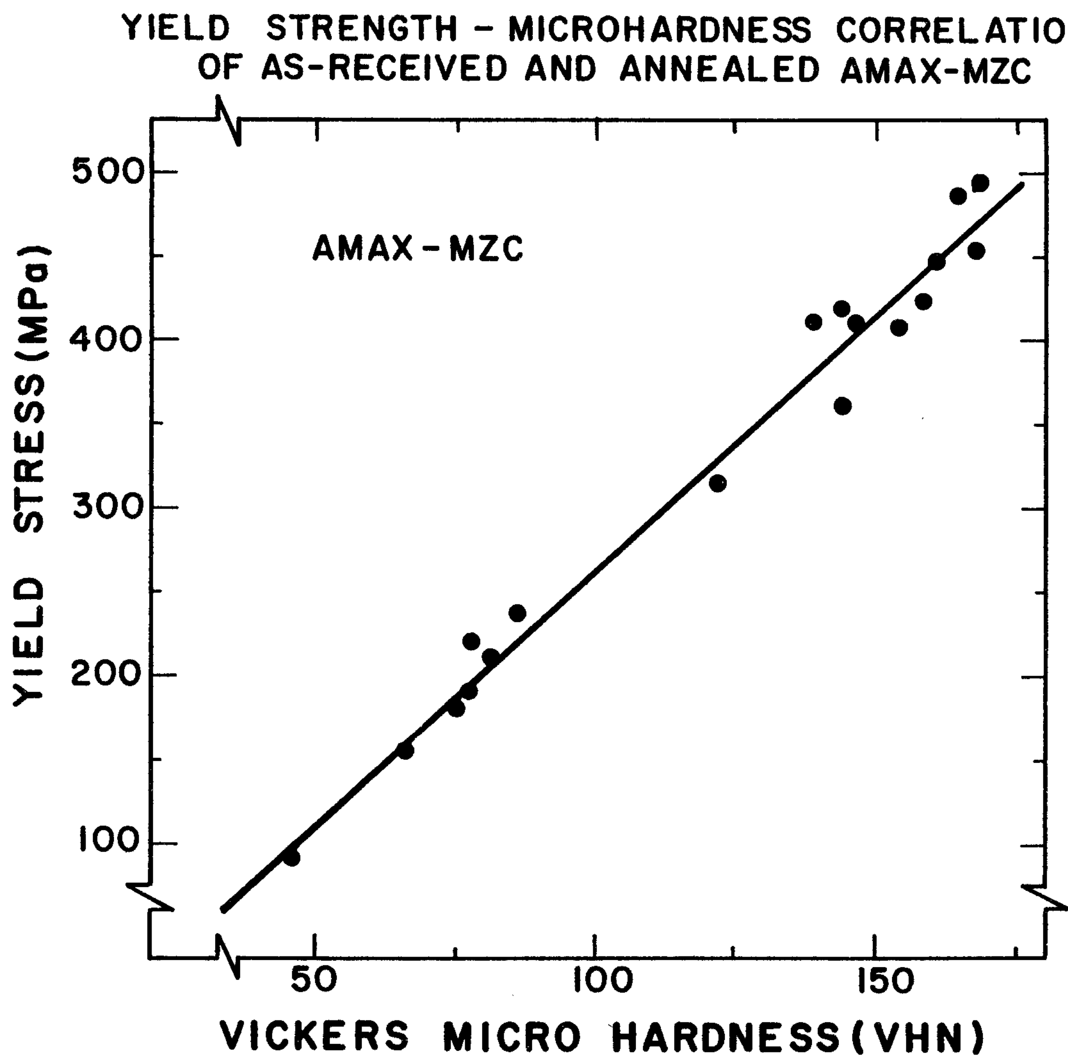


Fig. 6. Yield strength - microhardness correlation plot for CWA and annealed AMAX-MZC.