Influence of Thermomechanical Processing on the Microstructure and Properties of a Cu-Cr-P Alloy

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The microstructure and properties of a Cu-0.55 wt.% Cr-0.07 wt.% P alloy were studied by using optical microscopy, transmission electron microscopy, and the measurements of Vickers hardness and electrical conductivity after it was subjected to conventional aging and two thermomechanical treatments. The hardness increment resulting from the thermomechanical treatments was 50% higher than the increment produced by conventional aging. The thermomechanical procedure, including two aging steps (double aging), produced a 5% International Annealed Copper Standard (IACS) higher increase in conductivity than the procedure including a single aging step. However, the former procedure did not lead to more efficient hardening in the studied alloy than the latter procedure because during the second aging step extensive recovery or even the onset of recrystallization tended to suppress precipitation hardening to some extent. After being subjected to a thermomechanical treatment that included three cold-drawing steps and one aging step, the studied alloy showed a tensile strength of 550 MPa with a conductivity of 74% IACS. Based on the obtained results as well as on the comparison with other Cu-Cr type alloys, some suggestions were given for improving the thermomechanical processing route of the studied alloy.

Keywords CuCrP alloys, electrical conductivity, hardness, precipitation, recrystallization, thermomechanical processing

1. Introduction

Conventional precipitation treatments consisting of solution annealing and aging treatments (SAs) are often incapable of yielding all desired properties in precipitation-hardenable alloys. It has long been realized that thermomechanical processing (TMP) combining conventional precipitation treatments with plastic deformation can lead to an optimized combination of the properties. Typically, a TMP procedure combines heat treatments with cold deformation as follows: solution treatment \rightarrow cold deformation \rightarrow aging treatment (SCA). From the point of view of hardening, the TMP procedure is expected to produce a complete precipitation prior to the onset of recrystallization in the deformed supersaturated alloy during subsequent aging. For this reason, the control of processing parameters, such as the amount of cold deformation, the aging temperature, and time of processing, are of particular importance in the SCA procedure.

Besides the application of prior deformation, a double-aging treatment also has been widely used to process many precipitation-hardenable alloys. The solution-treated alloy is aged first at a lower temperature and then at a higher temperature. The first aging results in a fine dispersion of Guinier-Preston (GP) zones, which can act as heterogeneous nucleation sites for precipitation during the second aging. Therefore, the double-aging treatment leads to a finer precipitate distribution than does a single-aging treatment.^[1]

Copper-chromium alloys are a group of high-strength and high-conductivity copper alloys that are widely used in applications such as welding wheels, switch gears, circuit breakers, and cable connectors. A considerable amount of research work has been carried out for characterizing the properties and mi-crostructures of age-hardened Cu-Cr alloys,^[2-6] and the TMP of Cu-Cr alloys also has been investigated by a few researchers.^[7-10] In recent years, some new Cu-Cr-type alloys have been developed by adding trace amounts of a second alloying element (e.g., P or Fe).^[11-13] Our recent study^[11] on a Cu-0.61 wt.% Cr-0.07 wt.% P alloy showed that the precipitates formed in the overaged alloy consist mainly of Cr with a body-centered cubic (bcc) structure and that the alloy has a higher electrical conductivity and a stronger resistance to overaging than Cu-Cr binary alloys during conventional aging. In our earlier study, however, the alloy was not subjected to TMP. Moreover, no literature has so far been published on the TMP of the Cu-Cr-P-type alloys. Therefore, there is a need for understanding the interactions between the deformation and the precipitation of Cu-Cr-P-type alloys.

In the present study, the SCA procedure was applied to a copper alloy with the composition of Cu-0.55 wt.% Cr-0.07 wt.% P. Another TMP procedure, a double-aging treatment including a cold deformation step between two aging steps, also was used to process the studied alloy. The study first focused on the influences of the two TMP procedures on the microstructure, the hardness, and the electrical conductivity of the studied alloy. Then the alloy properties produced by a multistep TMP route were compared with the properties of other Cu-Cr-type alloys. The main objective of the study was to understand the relationship between the alloy properties and the processing steps of the TMP procedure in order to obtain useful information for the further development of Cu-Cr-P-type alloys.

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2. Experimental Procedures

Test material with the composition of Cu-0.55 wt.% Cr-0.07 wt.% P was produced from pure elements by vacuum melting and casting at Outokumpu Poricopper (Oy, Finland). After undergoing homogenization treatment for 30 min at 1050 °C, cast ingots with an 80 mm diameter were hot-extruded into rods with a 16 mm diameter.

Specimens cut from the hot-extruded rods were subjected to the following procedures:

- SA procedure (conventional aging): solution treatment for 60 min at 1000 °C → quenching in water → aging at 520 °C for times ranging from 15-120 min;
- SACA procedure (double aging): solution treatment for 60 min at 1000 °C → quenching in water → aging for 30 min at 450 °C → cold compression by 30% → aging at 520 °C for times ranging from 15-120 min; and
- SCA procedure: solution treatment for 60 min at 1000 °C
 → quenching in water → cold compression by 30% → aging at 480 °C for times ranging from 15-120 min.

Vickers hardness was measured using a Zwick hardness tester (Zwick GmbH & Co., Ulm, Germany) with a 5 kg load. Electrical conductivity was measured by a Sigma tester (Foerster UK Ltd., Tamworth, UK). The compressive deformation included in the SACA and SCA procedures was carried out using a Zwick universal materials testing machine (model 148700, Zwick GmbH & Co.). An optical microscope (Union Versamet 3, Union Optical Co. Ltd., Japan) equipped with a digital camera and a JEOL JEM-2010 analytical transmission electron microscope (TEM) with an acceleration voltage of 200 KV (Japan Electron Optics Ltd., Tokyo, Japan) were used for characterizing the heat-treated and deformed specimens. Thin foil samples for TEM were prepared by a single-jet electropolisher (model 550, Southbay Technology, San Clemente, CA) in a bath containing a solution consisting of 1 part nitric acid and 2 parts methanol at a temperature below -40 °C.

3. Results and Discussion

3.1 Microstructure After TMP

Figure 1 shows optical micrographs of the as-aged microstructures of the studied alloy corresponding to the SA, SCA, and SACA procedures. The structure resulting from the SA procedure (conventional aging) consists of large equiaxed grains and many uniformly distributed particles (Fig. 1a). Our earlier study^[11] showed that such particles are Cr_3P with a body-centered tetragonal (bct) crystal structure and that they are formed during casting and remain undissolved in the solution-treated state.

The aging step of the SCA procedure did not significantly change the structural characteristics caused by prior deformation; the grains shown in Fig. 1(b) were mainly flattened by compression and aligned in a direction perpendicular to the compression axis. There exist also many "particles" in the structure, but their distribution is not as uniform as the undissolved particles shown in Fig. 1(a). The larger magnification view in Fig. 1(b) reveals that many of the particles are recrystallized small grains that nucleated mostly at the subgrain and grain boundaries during the aging process (Fig. 1c). In contrast to the SCA procedure, the second aging in the SACA procedure resulted in more extensive recrystallization in the studied alloy. The new grains shown in Fig. 1(d) appear to be larger and more numerous than the recrystallized grains shown in Fig. 1(c).

Figure 2 presents TEM micrographs showing the asdeformed microstructure of the studied alloy and its microstructure processed by deforming and aging for 60 min at 480 °C (the SCA procedure). The compressive deformation introduced a high density of dislocations into the supersaturated solid solution (Fig. 2a). Dislocation distribution is not homogeneous, and it consists of a cellular substructure. Aging for 60 min at 480 °C caused an extensive rearrangement of dislocations so that the parallel subgrain boundaries are clearly visible (Fig. 2b). This indicates that in the specimen the polygonization has developed to a great extent due to extensive recovery. Despite there being no evidence of recrystallization revealed in the specimen that was aged for 60 min, recrystallization would start with increasing aging time, as shown in Fig. 1(c).

Figure 3 is a group of TEM micrographs showing singleaged and double-aged microstructures yielded by the SACA procedure. Figure 3(d) is the selected area diffraction pattern (SADP) recorded from Fig. 3(c). The first aging produced fine precipitates along with some curved dislocations in the vicinity of grain boundaries (Fig. 3a). Since the precipitates are so fine and densely distributed, it is difficult to measure their size. Our earlier study^[11] showed that quenching in cold water from the solution annealing temperature of 1000 °C introduces a lot of dislocations in the vicinity of the grain boundaries. The dislocation density shown in Fig. 3(a) has been significantly reduced from the solution-annealed and quenched states due to the aging.

After the specimen was aged for the second time (120 min at 520 °C) after the deformation, it showed a cellular substructure retaining some of the deformed characteristics. However, there was a dramatically reduced dislocation density inside many cells (Fig. 3b). Figure 1(d) is an optical micrograph of the same specimen shown in Fig. 3(b). The two figures suggest that recrystallization was started in the specimen but that it did not proceed to a great extent. A larger magnification view of Fig. 3(b) revealed the presence of precipitates inside the cells (Fig. 3c). The average particle size was estimated to be about 10 nm. Differing from the fine precipitate distribution shown in Fig. 3(a), the precipitates obtained by the second aging showed an increased particle size and interparticle spacing. This implies that the capability of the precipitates to inhibit recrystallization would degrade with their coarsening during the second aging. The satellite spots originating from the precipitate diffraction surround the main spots (Fig. 3d), indicating that there is a regular orientation relationship between the precipitates and the copper matrix.

Both of the specimens subjected to the SCA and SACA procedures retained their deformed characteristics after being aged for 120 min at 480 and 520 °C, respectively. However, they started to recrystallize, and the double-aging treatment (SACA procedure) resulted in a larger degree of recrystalliza-



Fig. 1 Optical micrographs showing the microstructures of the Cu-0.55 wt.% Cr-0.07 wt.% P alloy processed in different ways: (a) solution annealed and aged at 520 °C for 120 min; (b) solution aged for 120 min at 480 °C according to the SCA procedure; (c) a larger magnification view of (b); and (d) solution double aged for 120 min at 520 °C according to the SACA procedure

tion than the single-aging treatment (SCA procedure). The alloy specimen subjected to the SACA procedure was colddeformed after being first being aged at 450 °C. During the deformation, the pinning effect of precipitates can lead to a significant multiplication of dislocations so that the specimen contains a higher density of dislocations compared with the specimen deformed directly after solution treatment. The deformed alloy with a higher density of dislocations has more potential to recrystallization due to the higher driving force.^[14] The precipitates formed during the first aging can act as the barriers that retard the recrystallization. However, they would coarsen and have increased interparticle spacing with increasing time during the second aging, which decreases their capability of retarding the recrystallization. In addition, the second aging was carried out at a temperature (520 °C) that was higher than the aging temperature (480 °C) of the SCA procedure. This can be another reason for the more extensive recrystallization occurring in the specimen that was subjected to the second aging of the SACA procedure. Thus, the studied alloy is more susceptible to recrystallization during the second aging in the SACA procedure.

3.2 The Variations of Hardness and Conductivity During TMP

Figure 4(a) shows the variations of hardness and electrical conductivity obtained through the different steps of the SCA procedure. Both hardness and electrical conductivity were low after solution treatment. The hardness increased dramatically, whereas the conductivity decreased slightly due to the subsequent compression. Aging for 30 min at 480 °C caused a pronounced increase in both hardness and electrical conductivity. While the prior deformation introduced a high density of dislocations into the solution-treated alloy (Fig. 2a), it did not decrease the electrical conductivity significantly. This suggests that the low conductivity can be attributed mainly to the solute atoms dissolved by the solution treatment, and partly to the dislocations produced by the deformation.

During the SACA procedure, the first aging increased both electrical conductivity and hardness considerably (Fig. 4b). The subsequent compression further hardened the alloy but resulted in a decrease in conductivity of about 4% of the In-



Fig. 2 TEM micrographs showing (a) the as-deformed and (b) the deformed-plus-aged (60 min at 480 °C) microstructures of the Cu-0.55 wt.% Cr-0.07 wt.% P alloy that was subjected to the SCA procedure

ternational Annealed Copper Standard (IACS). One reason for the decrease in conductivity seems to be the high density of dislocations introduced by the subsequent deformation. A large amount of cold deformation is usually considered to have only a small influence on the conductivity of pure copper. For example, Ref. 15 reported that a decrease of only 2-3% IACS is caused by cold-working copper to a 90% reduction in thickness. In the present study, the alloy was cold-deformed by only a medium amount (30%). Thus, the abnormal decrease in conductivity due to the cold deformation may not result merely from the dislocations. Besides the influence of dislocations, there should be another reason for the decrease in the conductivity of the studied alloy.

It has been found^[9,10,16] that cold deformation following a precipitation treatment may extensively shear very fine precipitates (clusters) and may redistribute some solute atoms from them back to the copper matrix. Fargette^[9] reported that many precipitation-treated copper alloys, such as Cu-Ti, Cu-Zr, Cu-

Fe, Cu-Co, and Cu-Cr, show an abnormally large increase in resistivity after subsequent cold work. Szablewski and Haimann^[10] studied the resistivity of Cu-Cr binary alloys that were subjected to TMP. They reported that the deformation-induced dissolution of Cr solute atoms resulted in a large increment in resistivity after aging and cold working, and that the effect is severe in the early stage of the precipitation process, especially when chromium precipitate particles are smaller than 5 nm. As shown in Fig. 3(a), the fine precipitates resulting from the first aging are most possibly disturbed by subsequent deformation so that a part of the solute atoms redissolve into the matrix and scatter the conduction electrons. Although the resulting conductivity of the studied alloy is thereby reduced to some extent, it is still much higher than that in the solution-treated state and can also readily be raised again by the second aging (Fig. 4b).

Figure 5 illustrates the maximum hardness increases obtained by conventional aging as well as by different steps of the SCA and SACA procedures. The combination of the cold work and aging steps (SCA and SACA procedures) resulted in a total of at least 30 Vickers Hardness (HV) higher hardness increment than with conventional aging. However, it is noted that the maximum hardness increase arising from the aging step of the SCA procedure appears to be smaller than that resulting from conventional aging. Cold deformation after solution treatment leads to a deformed supersaturated solid solution. During subsequent aging, the processes of precipitation, recovery, and recrystallization may occur simultaneously and may interact with each other. The hardness increment due to aging does not result simply from precipitation hardening but rather from a consequence of the interactions of these processes. The present result implies that extensive recovery or even recrystallization may start with the progress of precipitation during the subsequent aging (see Fig. 1c), which partly eliminates the hardening of the predeformed alloy. To avoid the loss in hardening, the use of a lower aging temperature (e.g., 450 °C) in the SCA procedure is suggested, especially for the heavily predeformed alloy.

Figure 5 also shows that the SACA procedure produced only a slightly higher total hardness increase than did the SCA procedure. The second aging of the SACA procedure, in particular, resulted in a fairly small increase in hardness compared with the first aging. As discussed before, the deformation step of this procedure may disintegrate fine precipitate particles and may redistribute the solute atoms from the precipitates back to the matrix so that the disturbed precipitates cannot effectively retard the mobility of dislocations and prevent recovery and recrystallization during the second aging. Since the deformed alloy was aged a second time at a temperature (520 °C) that was higher than the first aging temperature, extensive recovery would occur, and recrystallization would even start, with increasing aging time. Meanwhile, the precipitation may be accelerated into the regime of overaging during the second aging, as the high density of dislocations offers preferred nucleation sites for precipitates and easy paths for diffusion.^[17,18] These processes can suppress the hardness increase that is caused by the second aging.

Figure 6 presents the hardness and electrical conductivity of the studied alloy as a function of aging time during the aging step of the SCA procedure and during the second aging



Fig. 3 TEM micrographs showing the as-aged structures of the Cu-0.55 wt.% Cr-0.07 wt.% P alloy developed by the SACA procedure: (a) first-aged structure containing a few curved dislocations near a grain boundary; (b) second-aged structure (120 min at 520 °C); (c) precipitate particles visible in the larger magnification view of the circled part in (b); and (d) SADP recorded from (c) showing satellite spots along with the main spots (face-centered cubic (fcc) Cu with the pattern of $[\bar{1}14]$ zone axis)

(double-aging) step of the SACA procedure. For comparison, Fig. 6 also shows the behavior of the hardness and electrical conductivity as a function of aging time when the studied alloy was conventionally aged at 520 °C. Evidently, both the SCA and SACA procedures produced a generally higher hardness than did conventional aging. The SACA procedure resulted in the highest hardness after a short duration of double aging. With increasing aging time, however, it produced a lower hardness than the aging step of the SCA procedure. During double aging, the existing unsheared precipitates coarsen readily through the easy diffusion paths provided by the high density of the dislocations. Therefore, the precipitation hardening in the alloy becomes less efficient due to the overaging effect with increasing aging time. At the same aging time, the double-aged specimen exhibits more extensive recrystallization than those subjected to single aging (Fig. 1c and d); the recrystallization softens the studied alloy more significantly with increasing aging time. For these reasons, the prolonged double aging of the SACA procedure produced less efficient hardening than the same aging time used in the SCA procedure.

The electrical conductivity of the studied alloy has a different response to increasing aging time than does hardness. The conductivity produced by double aging was about 5% IACS higher than that produced by the same aging time in the SCA procedure (Fig. 6). The second aging in the SACA procedure leads to a reduction in the density of dislocations, and, more importantly, it continues to decrease the number of solute atoms that are dissolved in the matrix. For these reasons, double aging is beneficial for electrical conductivity. However, the SACA procedure did not harden the studied alloy more efficiently, and, in practice, it is also more complicated than the SCA procedure.



Fig. 4 Changes in the hardness and electrical conductivity of the Cu-0.55 wt.% Cr-0.07 wt.% P alloy during different steps of (**a**) the SCA procedure and (**b**) the SACA procedure



Fig. 5 Maximum hardness increments of the Cu-0.55 wt.% Cr-0.07 wt.% P alloy as produced by the different steps of the SA, SCA, and SACA procedures

As is also shown in Fig. 6(b), conventional aging rapidly produced a maximum level in electrical conductivity, but after that the electrical conductivity was slightly lower at an aging time of 30 min. The large increase in conductivity is primarily due to the precipitation process, which drains the dissolved solute atoms from the copper matrix by forming the precipi-



Fig. 6 The hardness and electrical conductivity of the Cu-0.55 wt.% Cr-0.07 wt.% P alloy as a function of aging time during the aging steps of the SA, SCA, and SACA (the second aging step) procedures

tates. However, densely spaced fine precipitates that are formed in the early stage of precipitation can appreciably scatter the conduction electrons, thus increasing the resistivity to some extent.^[19] The same anomalous increase in resistivity also was observed in the Cu-0.61 wt.% Cr-0.07 wt.% P alloy during conventional aging.^[11] It is worth noting that the SCA and SACA procedures prevent the anomalous increase in resistivity of have reported that an anomalous increase in resistivity occurs in a solution-treated and quenched Cu-0.6 wt.% Cr binary alloy in the aging temperature range from 350-450 °C, but that the anomalous increase can be eliminated by prior cold deformation.

The studied alloy also was subjected to the following TMP route: solution treatment \rightarrow hot extrusion \rightarrow first cold drawing (true strain $\varepsilon = 1.7$) \rightarrow aging for 30 min at 450 °C \rightarrow second cold drawing ($\varepsilon = 0.9$) \rightarrow third cold drawing ($\varepsilon = 1.8$). This TMP procedure produced a 550 MPa tensile strength and 74% IACS conductivity for the studied alloy.^[20] For comparison, Table 1 lists the combinations of tensile strength and corresponding electrical conductivity values for the studied alloy and other Cu-Cr-type alloys after being subjected to TMP procedures.

 Table 1
 Typical Combinations of the Tensile Strength and Conductivity Values for the Studied Alloy and Other
 Cu-Cr-Type Alloys

Alloy, wt.%	Condition	UTS, MPa	Cond, %IACS	Reference
Cu-0.55Cr-0.07P (a)	Deformation + aged (30 min / 450° C)(a)	550	74	21
Cu-0.7Cr-0.3Fe	90% CW + aged (90 min / 450°C)	537	66	13
Cu-0.28Cr-0.08Ti-0.05Si	80% CW + aged (60 min / 470°C)	560	78	23
Cu-0.65Cr	80% CW + aged (180 min / 425°C)	505	79	22
Cu-5.7Cr (b)	Deformation + aged (12 h / 420°C)(b)	925	72	24

(a) Include three cold deformation steps reducing the rod diameter from 19 to 2 mm (total true strain $\varepsilon_{\text{total}} = 4.4$). (b) Include three cold deformation steps reducing the rod diameter from 25 mm to 0.36 mm ($\varepsilon_{total} = 8.5$).

The studied alloy shows a better combination of tensile strength and electrical conductivity than the Cu-0.7 wt.% Cr-0.3 wt.% Fe alloy^[13]; the former would seem to have a higher conductivity than the latter if they both were similarly processed. The Cu-0.65 wt.% Cr alloy,^[21] which contains 0.1% more chromium than the studied alloy, shows a lower tensile strength but a slightly higher conductivity than the studied alloy. As compared with the studied alloy, the Cu-0.65 wt.% Cr alloy is aged for a longer time (180 min) after being deformed by 80%. The processing can result in a complete precipitation and can reduce the dislocation density to a great degree. Although containing less chromium, the Cu-0.28 wt.% Cr-0.08 wt.% Ti-0.05 wt.% Ti alloy^[22] shows a better combination of tensile strength and conductivity than both the Cu-0.55 wt.% Cr-0.07 wt.% P and the Cu-0.7 wt.% Cr-0.3 wt.% Fe alloys. This suggests that a combined addition of two trace-alloying elements may be useful for improving the properties of Cu-Crtype alloys. Of all the alloys listed in Table 1, the Cu-5.7 wt.% Cr alloy^[23] shows the highest tensile strength with a high conductivity (72% IACS). The high strength of the Cu-5.7 wt.% Cr alloy is yielded through a large amount of deformation (total true strain of 8.5), which produced very thin wires with a final diameter of 0.36 mm.^[23] It is interesting to note that neither a high content of chromium nor a large amount of deformation reduce the conductivity of the Cu-5.7 wt.% Cr alloy to a great degree. According to the earlier studies^[24,25] on the electrical conductivity of this kind of deformation-processed copper alloys, the elongated inclusion filaments (e.g., Cr or Nb) are like walls inside the copper matrix and subdivide it into pure copper cells, which act as "channels" for the transport of conduction electrons.

As compared with the conventional aging that produced a maximum onductivity of 93% IACS (Fig. 6), the multistep thermomechanical route resulted in an almost 20% reduction in the conductivity of the studied alloy. Based on the results and discussion presented before, some suggestions can be given for improving the TMP route, as follows: (1) carry out the aging treatment after the second cold drawing; (2) use a smaller amount of deformation (e.g., $\varepsilon = 0.9$) in the final cold drawing; (3) use a low aging temperature (e.g., 425 °C); and (4) prolong the aging time to 60 min. By making these changes, the disintegration of precipitate particles due to cold working may be avoided to some extent; the number of solute atoms dissolved in the copper matrix can be further reduced with extended aging time. Therefore, it is very likely that the new TMP route will lead to a better combination of strength and electrical conductivity in the studied alloy.

4. Conclusions

The following conclusions can be drawn from the results of the study on the TMP of the Cu-0.55 wt.% Cr-0.07 wt.% P alloy.

- The hardness increments produced by the SCA and SACA procedures are at least 50% higher than those obtained by conventional aging. The SACA procedure generally yields almost 5% IACS higher conductivity than the SCA procedure. However, the SACA procedure has no obvious advantage in hardening the studied alloy and, practically, is more complicated than the SCA procedure.
- During the aging of deformed supersaturated alloys, the onset of recrystallization and the accelerated overaging often lead to an inefficient hardening. Therefore, it is particularly important to use proper processing parameters (e.g., aging temperature and time) during TMP. In the SACA procedure, for example, first aging at 420 °C and second aging at 480 °C may further improve the hardening of the studied alloy.
- Compared with the pure copper that was cold-worked by 90%, the underaged Cu-0.55 wt.% Cr-0.07 wt.% P alloy shows a larger reduction in conductivity after being colddeformed by 30%. The deformation after aging treatment may disintegrate very fine precipitates and may induce partial redissolution of the solute atoms into the copper matrix.
- Both the SCA and SACA procedures eliminate the anomalous increase in the resistivity of the studied alloy, which was observed under the conventional aging condition, nearly producing the peak hardness.
- The studied alloy shows a tensile strength of 550 MPa, with a conductivity of 74% IACS after being processed by a TMP route including three cold-drawing steps and one aging step. Most likely, the strength and conductivity of the studied alloy can be improved further by modifying the TMP route.

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