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Ductilization of Cr via Oxide Dispersions

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Summary:

Work by Scruggs et al. in the 1960's demonstrated that up to 20% tensile ductility could be achieved at room-temperature in sintered and extruded powder metallurgical Cr alloyed with MgO. During sintering, much of the MgO converts to a $MgCr_2O_4$ spinel, which was hypothesized to getter nitrogen from the Cr, rendering it ductile. Recent efforts at Oak Ridge National Laboratory (ORNL) have succeeded in duplicating this original effect. Preliminary results suggest that the ductilization mechanism may be more complicated than the simple nitrogen gettering mechanism proposed by Scruggs, as some ductility was observed at room-temperature in Cr-MgO alloys containing nitride precipitates. Results of microstructural characterization and room-temperature mechanical property studies are presented for Cr-6MgO-(0-2.2)Ti wt.% as a function of hot-pressing and extrusion. Possible mechanisms by which the MgO additions may improve the room-temperature ductility of Cr are discussed.

Keywords:

chromium, magnesia, spinel, mechanical properties, ductility, interstitial embrittlement, powder processing

1. Introduction

Chromium metal possesses an attractive combination of properties such as high melting point, moderate density, good high-temperature oxidation resistance, and excellent low- and high-temperature corrosion resistance in many environments (1). However, its brittle to ductile transition temperature (BDTT) is usually, but not always, above room-temperature and, further, it is susceptible to environmental embrittlement at elevated-temperatures by interstitial nitrogen penetration (2,3). Therefore, industrial uses of Cr as a structural material have been limited to low impact/non critical loading applications.

The development of Cr as a structural material was aggressively pursued in the 1960's (3). One of the most interesting findings from this era was pioneering work by Scruggs et al. (4-7), who discovered that the room-temperature tensile ductility of Cr could be significantly improved by the addition of MgO. Blended, sintered, and extruded powders of commercial purity Cr, MgO, and Ti in the range of Cr-(2-6)MgO-0.5Ti weight percent (wt.%) were reported to exhibit room-temperature plastic tensile elongations of up to 20% in the as-recrystallized state, and without electropolishing. The material was successfully used for components such as a gas turbine flame holder and as a thermowell in an ethylene cracking furnace. However, efforts to create environmental embrittlement-resistant, high-strength Cr (and Cr-MgO) alloys for demanding high-temperature applications such as gas turbine blades, with BDTT's below room temperature, were unsuccessful (3). Interest in Cr as a high-temperature structural alloy waned in the 1970's, and the work by Scruggs et al. has been largely ignored since then.

There has been a recent resurgence of interest in Cr as a structural material (8-13), driven by its excellent high-temperature corrosion resistance, which makes it of great utility for the chemical and process industries, and its relatively low coefficient of thermal expansion (CTE), which is attracting attention as a metallic interconnector in solid oxide fuel cells (14). Improvement in room-temperature mechanical properties is still a major goal. The driving force behind the present work has been to gain a fundamental understanding of why MgO additions to Cr improve ambient temperature ductility, and to revisit the possibility of developing Cr-MgO alloys for moderate impact/load- bearing structural applications in hot, aggressive environments. Applications of interest include nozzles, spouts, and brackets in black liquor gasification environments (15). Here we present preliminary results from microstructural and mechanical characterization studies of the original Scruggs Cr-6MgO-0.5Ti wt.% alloy, and from our initial attempts at fabricating ductile Cr-MgO base alloys.

2. Characterization of Scruggs Cr-6MgO-0.5Ti wt.% Alloy

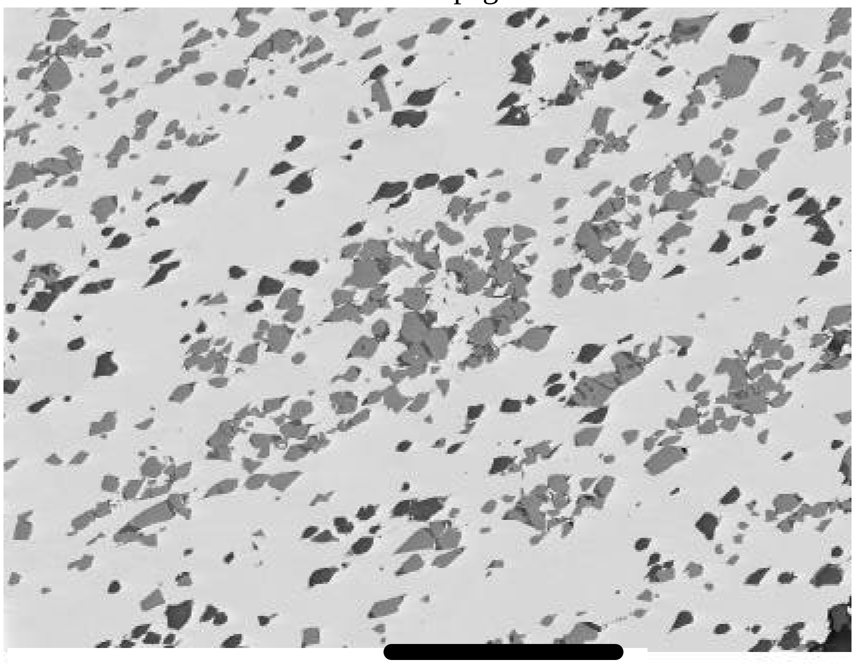
A major contributor to the ambient brittleness of Cr is the presence of tramp interstitial elements (nitrogen, carbon, etc.), which can result in the formation of acicular phases at the grain boundaries (1-3). The ductilizing mechanism proposed by Scruggs was based on in-situ gettering of interstitial impurities, primarily nitrogen. During sintering, much of the MgO added to commercial purity Cr powder converts to the $MgCr_2O_4$ spinel phase, which Scruggs hypothesized gettered nitrogen and other impurities from the Cr, rendering it ductile (5,6). High levels of oxygen impurities in the Cr powder (0.5-0.6 wt.%) were considered necessary to form the $MgCr_2O_4$ spinel phase (5). The Ti was added for general gettering purposes.

Table 1- Chemical analysis (inductively coupled plasma and gas fusion) of the Scruggs Cr-6MgO-0.5Ti alloy. Data for a typical hot-pressed Cr-MgO-Ti alloy of the present work, as well as for the source Cr and MgO powders, are also shown. All compositions are reported in wt.%.

Element	Scruggs Cr-6MgO-0.5Ti	Cr-6MgO-1Ti	Cr Powder	MgO Powder
Cr	92.9	92.05	99.17	-
Mg	3.33	3.89	-	64.88
Ti	0.48	1.08	-	-
O	3.116	2.7	0.59	33.82
N	0.031	0.038	0.025	<0.1
C	0.04	0.06	0.01	0.11
S	0.019	0.011	0.02	0.12
Al	0.01	-	0.01	0.89
Si	0.02	0.07	0.01	0.04
Fe	0.04	0.1	0.09	0.03
Ca	0.01	-	-	0.1
W	-	-	0.06	-
Ni	-	-	0.01	-
Na	-	-	-	0.01

An ingot of Cr-6MgO-0.5Ti wt.% alloy manufactured in the 1960's was obtained from Dr. Scruggs for study. Details of the processing conditions for this specific ingot could not be found; however these alloys were generally manufactured by blending of commercial purity powders, sintering at approximately 1600°C for 1-2 h, and then extruding at 1200°C (typically at a 9:1 ratio) (4,5). A chemical analysis of this alloy is presented in Table 1. The alloy contained relatively high levels of carbon (0.04 wt.%), nitrogen (0.031 wt.%), and sulfur (0.019 wt.%), which is expected given the use of commercial-purity Cr powder.

The microstructure of the Cr-6MgO-0.5Ti alloy consisted of a nominally pure chromium matrix with aligned bands of oxide particles of two distinct types: magnesium oxide and magnesium-chromium-titanium oxide (Fig. 1).



MgO (dark particles) 100 µm
2 $MgCr_2O_4$• Mg_2TiO_4 Spinel Phase (gray particles)

Fig. 1 - SEM cross-section micrograph of Scruggs Cr-6MgO-0.5Ti wt.% alloy. Spinel phase identification based solely on composition.

In particular, no titanium was detected in the matrix phase. Electron probe microanalysis (EPMA) of the complex oxide, quantified using pure element standards for Cr, Mg and Ti, Al_2O_3 for oxygen and BN for nitrogen, indicated a composition (in wt.%) of 15Mg-40Cr-10Ti-35O. To within the experimental uncertainty in the measurement, this composition corresponds to a spinel on the $MgCr_2O_4$-Mg_2TiO_4 tie line, of composition 2 $MgCr_2O_4$ • Mg_2TiO_4. No chromium nitrides were detected by EPMA. The EPMA results indicated some nitrogen (0.1-0.5 wt.%) in the spinel phase; however, given the peak overlaps between the N-K and Ti-L lines, the presence of nitrogen in the spinel cannot be unambiguously identified even with wavelength-dispersive spectroscopy (WDS). Electron energy-loss spectroscopy (EELS) in the transmission electron microscope (TEM) is planned to unambiguously determine the presence or absence of nitrogen in the spinel (see section 3).

The hardness of the Scruggs alloy was in the range of 150-180 Vicker's (VHN), which is consistent with a recrystallized structure (as reported by Scruggs). Tensile properties were evaluated using dogbone-type samples approximately 3.8 cm long, with a gage length of 1.27cm and a thickness of 0.6-0.7 mm. The surfaces were abraded to a 600 grit (United States standard) finish. The tensile testing was conducted in humid laboratory air using a crosshead speed of 2.54 mm/min, which yielded a strain rate of approximately 3.33×10^{-3}/s. Despite a nitrogen impurity level of 0.031 wt.%, the alloy

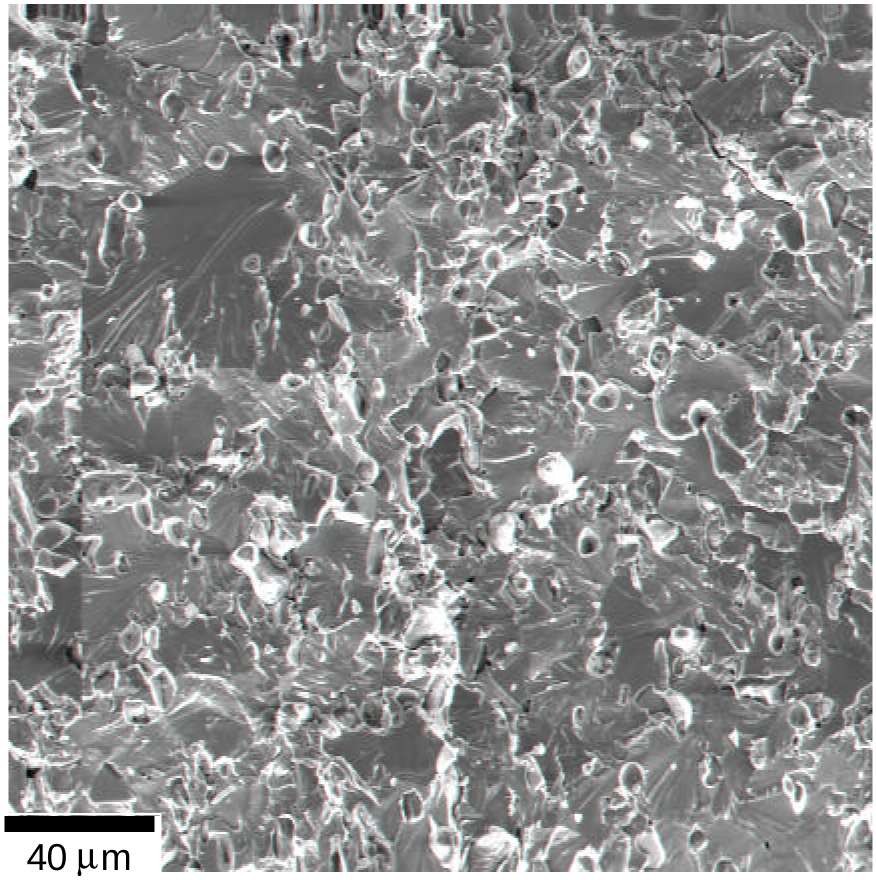


Fig. 2- SEM fractograph of tensile dogbone sample of Scruggs Cr-6MgO-0.5Ti wt.% alloy shown in Fig 1.

exhibited substantial plastic tensile elongation at room-temperature, ranging from 6.9–10.7% (Table 2). For pure Cr (without MgO additions) to exhibit appreciable room-temperature ductility at these strain rates, it needs to be very high purity (< 0.001-0.005 wt.% nitrogen), usually (but not always) cold-worked, and the surfaces electropolished to eliminate local notches (1,2).

Table 2 - Room-temperature tensile properties for Scruggs sintered and extruded Cr-6MgO-0.5Ti wt.% alloy.

	Tensile Strength MPa		
Finish	Yield	Fracture	% Elongation
600 grit (U.S.)			
	240	314	6.9
	246	328	6.9
	253	358	10.7

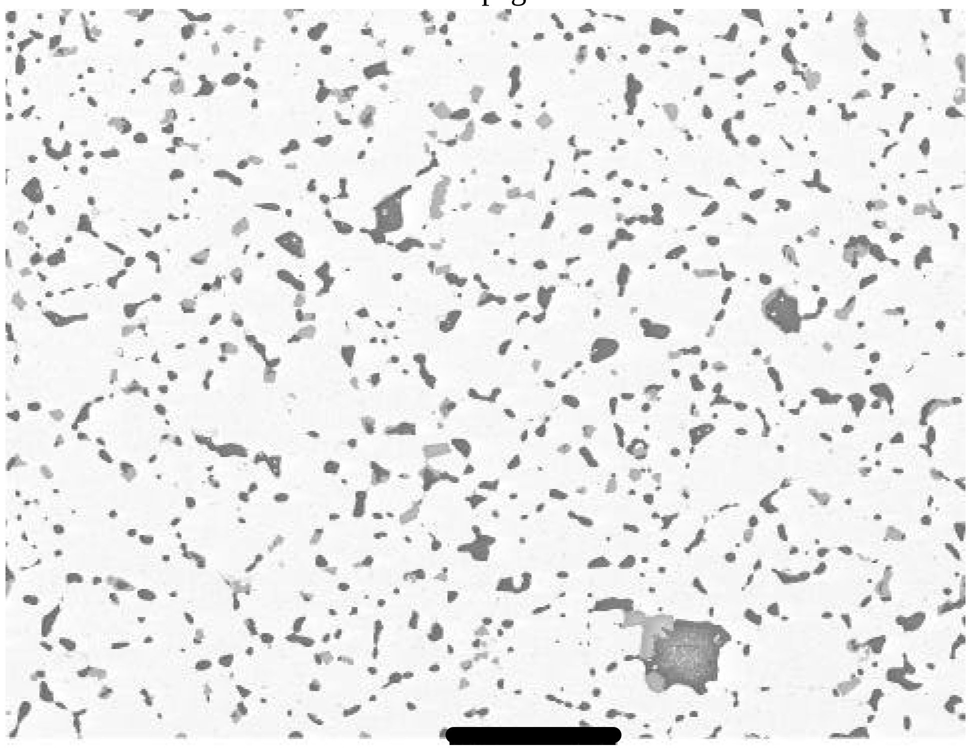
When the Cr-6MgO-0.5Ti alloy was polished to a coarse 180 grit finish, the plastic tensile elongation was reduced to below 2%. Therefore, the MgO addition decreased, but did not completely eliminate, notch sensitivity. The fracture of the alloy was predominately cleavage-type in all cases, regardless of the surface finish or the extent of plastic tensile elongation (Fig. 2).

3. Initial Attempts at Fabricating Cr-MgO Based Alloys

A series of Cr, Cr-0.5Ti, Cr-6MgO, and Cr-6MgO-(0.5, 0.75, 1, 2.2)Ti wt.% alloys was made in order to investigate the ductilizing effect of MgO on Cr. Commercial purity Cr, Ti, and MgO powders of nominal 1-5 μm size range were used. Chemical analysis of the Cr and MgO powders, as well as a typical Cr-MgO-Ti alloy that was made, Cr-6MgO-1Ti wt.%, are shown in Table 1. The MgO powder was calcined and stored in argon to prevent interaction with water vapor. Unless otherwise noted, the alloys were produced by oblique blending the powders for 24h using zirconia balls, and hot-pressing under vacuum in a graphite die at 1590°C for 2 h at a load of approximately 20 MPa. Selected alloys were further processed by sealing the hot-pressings in a steel can and extruding at 1300°C with a 9:1 reduction ratio. Both hot-pressing and hot-pressing/extrusion yielded fully dense material. The hardness of the hot-pressed and hot-pressed and extruded Cr-6MgO-(0-1)Ti wt.% alloys was in the range of 150-180 VHN, similar to the Scruggs alloy and, again, consistent with a recrystallized structure.

The microstructure of hot-pressed Cr-6MgO is shown in Fig. 3. The alloy grain boundaries were decorated by MgO and Mg-Cr-O phases. The composition of the ternary oxide phase was determined by EPMA to be 12Mg-52Cr-36O (wt.%), consistent with the $MgCr_2O_4$ spinel phase. Within the sensitivity limits of EPMA, nitrogen was not detected in the spinel phase. A similar microstructure was observed for hot-pressed Cr-6MgO-0.5Ti and Cr-6MgO-1Ti, except that Ti was present in the spinel phase (similar to the Ti spinel segregation observed in the Scruggs Cr-6MgO-0.5Ti alloy).

The presence of Cr-nitrides in Cr-6MgO was detected by the scanning electron microscopy (SEM) technique of low-voltage EDS spectrum imaging (16). This technique provides an attractive alternative to EPMA for mapping of different phases through their characteristic X-ray spectra, although it is not as good for quantitative analysis. A low operating voltage is used relative to the EPMA, which results in an order of magnitude better spatial resolution (~150 nm for 4 kV); however, many of the X-ray lines most suitable for quantification (e.g., Cr-K) are not excited at these voltages. The much poorer spectral resolution of EDS relative to WDS also compromises quantitative compositional analysis. However, because a full spectrum, rather than the intensities of a few selected X-ray lines, are acquired at each pixel in the image, low-voltage EDS spectrum imaging provides a true phase, rather than elemental, mapping technique. In-house ORNL multivariate statistical analysis software is used to identify all spectrally-distinct phases in the image, and also robustly distinguishes phases having spectral overlaps (e.g., O-K and Cr-L) (17,18).



100 µm

MgO (dark particles)
$MgCr_2O_4$ Spinel Phase (gray particles)

Fig. 3 - SEM cross-section micrograph of hot-pressed Cr-6MgO wt.% alloy. Spinel phase identification based solely on composition.

A gray-scale representation of a 200 x 200 pixel spectrum image, acquired at 4 kV with 100 nm per pixel, is shown in Fig. 4 for Cr-6MgO. In addition to the Cr matrix, MgO and $MgCr_2O_4$ phases, chromium nitride and sulfide phases were also identified. The nitrides were typically located adjacent to the MgO and $MgCr_2O_4$ oxide dispersions. Qualitatively, the nitrides appeared more blocky than the acicular nitrides typically observed in pure Cr. Similar nitrides were also observed in hot-pressed Cr-6MgO-1Ti (the microstructure of hot-pressed Cr-6MgO-0.5Ti has not yet been examined for nitrides). Interestingly, preliminary analysis of the sintered and extruded Scruggs Cr-6MgO-0.5Ti alloy and a hot-pressed and extruded Cr-6MgO-0.75Ti alloy of the present study did not indicate any nitrides in the microstructure, although further work is necessary to definitively rule out their presence.

Room-temperature tensile properties of the hot-pressed and hot-pressed/extruded alloys are summarized in Table 3. All samples were tested under the same conditions as for the Scruggs Cr-6MgO-0.5Ti alloy (600 grit finish, strain rate of approximately 3.33 $\times 10^{-3}$/s). Both the pure Cr and Cr-

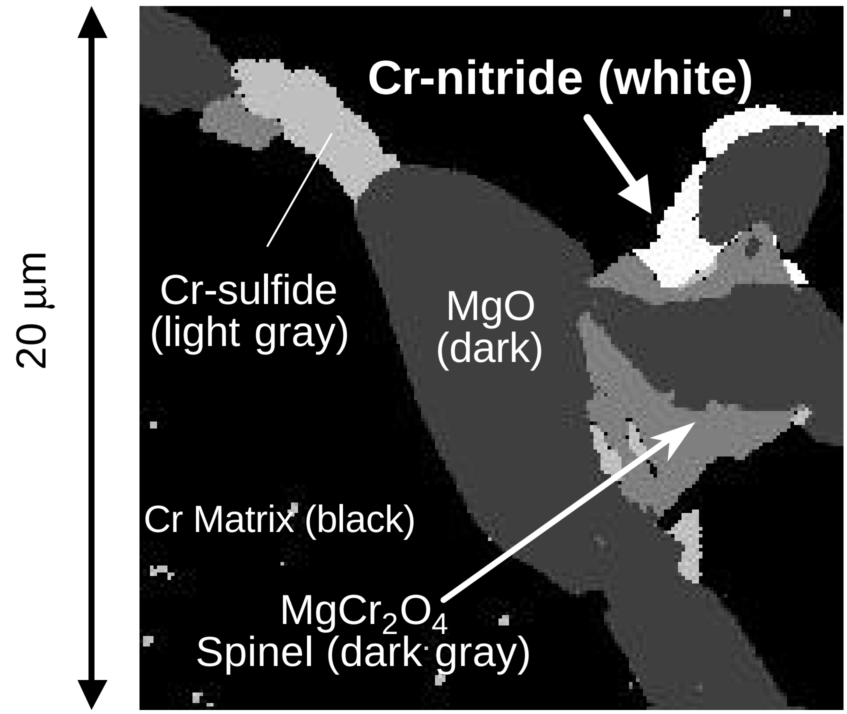


Fig. 4 -Phase map of the hot pressed Cr-6MgO specimen shown in Fig. 3, as acquired by low-voltage EDS spectrum imaging.

0.5Ti wt.% control alloys exhibited brittle behavior at room-temperature, with little plastic tensile elongation (<~2%) observed. However, plastic tensile elongation in the 3-5% range was observed for hot-pressed Cr-6MgO and Cr-6MgO-1Ti (Table 3), despite the presence of Cr nitrides (Fig. 4). (Similar ductility was also obtained for hot-pressed Cr-6MgO-0.5Ti). It should be noted that the grain size of the Cr and Cr-0.5Ti alloys was on the order of 0.5 millimeter while the grain size of the hot-pressed Cr-6MgO based alloys was on the order of 50 microns.

Increasing the Ti content to 2.2 wt.% in hot-pressed material resulted in a reduction in plastic tensile elongation to the 1% range, although the yield strength was increased from the 200 MPa range to over 300 MPa (Table 3). Subsequent extrusion of hot-pressed Cr-6MgO-0.75Ti (Fig. 5) yielded plastic tensile elongations over 10%, similar to that obtained for the Scruggs sintered and extruded Cr-6MgO-0.5Ti alloy (Tables 2, 3).

Table 3- Room-temperature tensile properties for selected alloys manufactured in the present work.

	Tensile Strength MPa		
Alloy (wt.%)	Yield	Fracture	% Elongation
Hot-Pressed 1590°C for 2 h			
Pure Cr			
	156	182	0.6
	149	202	1.1
*Cr-0.5Ti			
	178	229	1
	185	246	1.2
Cr-6MgO			
	220	296	3.4
	209	290	3.5
	205	310	4.9
	220	324	5.2
Cr-6MgO-1Ti			
	210	322	3.1
	175	322	4.7
	202	340	5.2
	200	350	5.4
Cr-6MgO-2.2Ti			
	333	358	0.6
	338	372	1.2
	283	338	1.2
Hot-Pressed 1590°C for 2 h and Extruded 1300°C 9:1 Ratio			
Cr-6MgO-0.75Ti			
	227	393	12.9
	243	388	10.7
*Hot isostatically pressed at 1600°C, 1.5 h, Nb can			

(a)

(b)

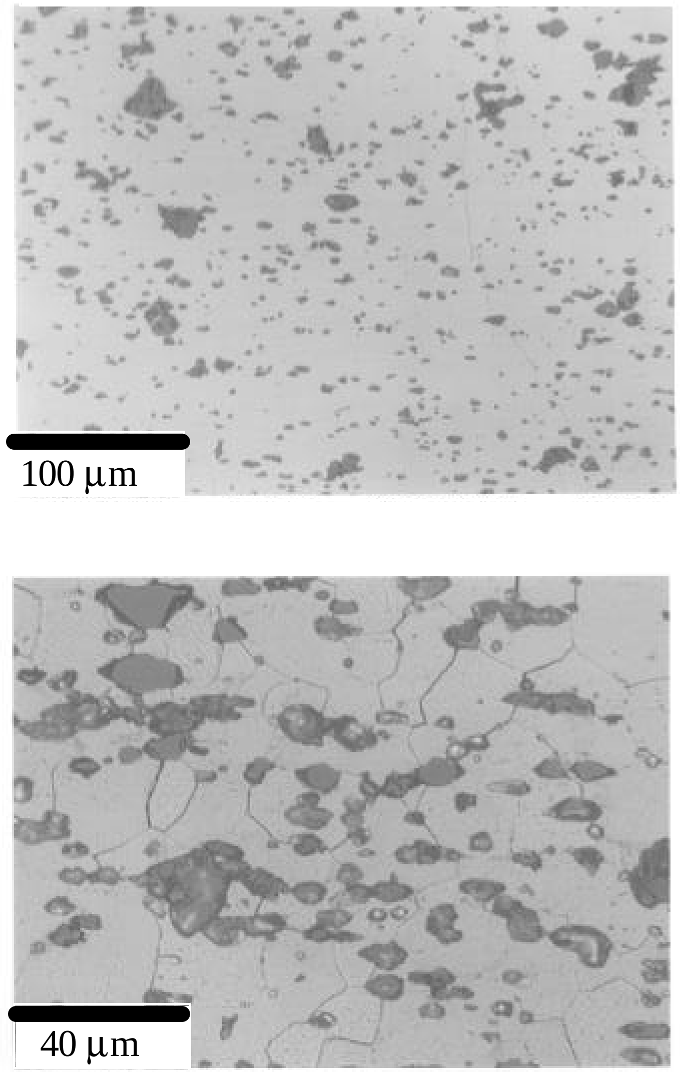


Fig. 5: Optical cross-section micrographs of hot-pressed and extruded Cr-6MgO-0.75Ti. a) longitudinal b) transverse (10% oxalic etch)

4. Discussion, Implications, and Planned Work

Overall, it was found possible to reproduce the beneficial effect of MgO on the room-temperature tensile ductility of Cr reported by Scruggs. The room-temperature tensile ductility of the Scruggs alloy and the hot-pressed alloys of the present work suggests that the addition of MgO to Cr significantly increases the tolerance of Cr to tramp interstitial impurities such as nitrogen and decreases, but does not completely eliminate, notch sensitivity. This latter finding is consistent with reports by Scruggs that, while the tensile BDTT of the Cr-MgO alloys was below room-temperature, the impact BDTT was above room-temperature (4,7). The addition of Ti in the 0-1 wt.% range to Cr-6MgO appeared to have little effect on room-temperature tensile properties (evaluated for hot-pressed material only).

The observation of Cr nitrides in the hot-pressed Cr-6MgO and Cr-6MgO-1Ti alloys was very surprising given that these alloys showed ~5% plastic tensile elongation at room-temperature (Table 3, Fig. 4). This result suggests that the ductilization of Cr by MgO is more complicated than the spinel nitrogen gettering mechanism proposed by Scruggs. Contributing factors to the ductilizing effect of MgO on Cr are speculated to include a switch to a more benign morphology of the Cr nitride phase and/or a shielding of its detrimental effects by its location adjacent to $MgO/MgCr_2O_4$ particles, and the production of a relatively fine-grained material (50 micron size range). Tucker et. al. (19) observed an effect of Cr_2N precipitate morphology on BDTT in pure Cr as a function of quenching and ageing treatments, although the results were not definitive. A reduction in grain size has been reported to increase low temperature ductility and fracture toughness in Cr (e.g. 1, 20). The introduction of fine particles such as MgO may also lower the BDTT by decreasing the effective slip plane length (reference 21 as cited in reference 22).

Attempts at introducing other ceramic dispersions in Cr have produced mixed results on room-temperature tensile ductility (3, 23-25) with no dispersions as effective as MgO. (Scruggs also demonstrated a beneficial effect with the direct addition of $MgAl_2O_4$ spinel to Cr (6), however, the available data suggest that it was not as effective at ductilizing Cr as MgO). The present authors have conducted preliminary investigations of additions of TiO_2 and Y_2O_3 hot-pressed under the same conditions as the Cr-MgO alloys, none of which resulted in significant room-temperature tensile ductility.

The factors contributing to the unique effect of MgO in ductilization of Cr (beyond its tendency to form a spinel phase with Cr_2O_3) are not clear. One quality of MgO is that it is quite soft for a ceramic phase and is therefore likely to be well tolerated by the Cr matrix during deformation. This may be necessary to realize the beneficial effects of grain size reduction resulting from the introduction of second phase particles in Cr. Second, as pointed out by Bakun et al. (22), MgO has a very high CTE relative to Cr, which likely results in high tensile stresses at the MgO-Cr interface. This could cause preferential precipitation of impurity phases at this location (22); Bakun et al. (22) also reported the presence of precipitated inclusions at the MgO-Cr interface similar to that observed in the present work for Cr-nitrides. Evaluation of the mechanical properties of Cr with ceramic dispersions of significantly higher CTE than Cr are planned to evaluate this mechanism.

In the present work, subsequent extrusion more than doubled the observed plastic tensile elongation at room-temperature in hot pressed material (Table 3). The reasons for this are not yet known. The increased ductility could be due to the texture introduced by the extrusion treatment (the tensile axis was aligned with the extrusion direction), a further reduction in grain size, or a more uniform distribution of $MgO/MgCr_2O_4$ particles. It could also result from complete removal of the nitride precipitates observed in hot-pressed material by effective capture by the $MgCr_2O_4$

spinel phase as a result of the driving force provided by the extrusion treatment. This latter explanation is effectively a refinement of the original mechanism proposed by Scruggs (5,6). However, it is important to stress that further characterization work is needed to confirm the absence of nitrides in extruded material and/or incorporation of nitrogen into the spinel phase. Critical experiments are also underway to determine the relative contributions of nitrogen management (altered nitride morphology and/or gettering by the spinel phase) and metallurgical effects (grain size refinement, texture, dispersed particle interactions with dislocations, etc.) to the ductilization effects of MgO on Cr.

An implication from all of the aforementioned proposed mechanisms is that mechanical properties may be improved if a finer particle dispersion can be achieved. This would reduce grain size and reduce the effective internal notch size, and possibly increase the efficiency of interstitial impurity management and gettering (if it does in fact occur). To accomplish this, work has been initiated to examine the effect of high-energy ball milling the Cr, MgO, and Ti powders prior to consolidation. Both the addition of MgO powder directly and the addition of Mg metal powder, with the goal of precipitating out fine MgO by internal oxidation, are planned.

5. Conclusions

1) The addition of MgO to Cr resulted in ~5% tensile plasticity in hot-pressed Cr-6MgO-(0-1)Ti wt.% and ~10% tensile plasticity in hot-pressed and extruded Cr-6MgO-0.75Ti at room-temperature, in an as-recrystallized state with a 600 grit surface finish and at a strain rate of approximately 3.33×10^{-3}/s.

2) The ductilization mechanism by which MgO acts is complex. In addition to apparent management of interstitial impurity elements such as nitrogen, through modification of nitride morphology and/or internal gettering, a significant contribution due to metallurgical factors such as yielding a fine grain size or a dispersed particle effect on dislocation behavior cannot be ruled out.

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