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Jung, C., Seraphin, S., and Raghavan, S., Department of Materials Science and Engineering, University of Arizona, Tucson, AZ 85721. TEM analyses of magnetic particles used in particulate recording media. High performance particulate magnetic recording media require consistent and uniform magnetic characteristics over the entire media surface. The properties of magnetic recording media can be influenced by the particle characteristics, such as shape, size and presence of defects. In this study, characterization of iron based metal particles (coercivity, H_c = 1500 Oe; magnetization, M_s = 120 emu/gm) and cobalt-modified iron oxide particles (H_c ~ 600 Oe, M_s ~ 75 emu/gm) were carried out using transmission electron microscopy (TEM).

In the case of cobalt-modified iron oxide particles, the effect of attrition milling in a liquid medium on particle characteristics was investigated. It was found from the micrographs that the acicular particles (length ~ 0.34 μm, aspect ratio ~ 7:1) were not damaged during the milling process. The micrographs of the particles also showed that there are some pores and irregularities at the surface. This may be due to the large material transport and the density change that take place in the solid-state reactions of the dehydration and reduction processes during particle synthesis.

Compared to the cobalt-modified iron oxide particles, the metal particles were smaller in size and had a more uniform surface. The length and aspect ratio of these particles were about 0.28 μm and 10:1, respectively. These particles contained an iron core of 120 Å in diameter with a 30 Å thick oxide layer. It was found that the thickness of the oxide layer was increased during the time of examination. This is probably due to the oxidation of the iron core by the high energy electron beam.

YOKOI, H., SERAPHIN, S., and KINGERY, W. D. Department of Materials Science and Engineering, University of Arizona, Tucson, AZ 85721. In situ investigation of sintering of alumina. Most academic studies of sintering phenomena have dealt with high temperature isothermal solid state processes. However, from the practical point of view, one of the most important aspects of sintering is microstructure changes occurring during the heating-up process. We find that shrinkage mainly occurs below the eutectic temperature in various liquid phase sintering systems. If lower temperature processes are important, the distribution of the additive should be critical. We have examined the sintering rate of the alumina-titania system. Three samples were heated at 1200°C, much below the eutectic temperature of this system, and quenched. One consisted of the pure Al_2O_3 powder prepared from ammonium hydroxy carbonate. A second consisted of the Al_2O_3 powder milled together with a TiO_2 powder additive. A third sample was made by a colloidal technique. Titanium isopropoxide was added to a suspension of the Al_2O_3 powder and hydrolyzed to form a fine coating on each particle. TEM studies have shown that the alumina particle surfaces act as sites for heterogeneous nucleation. The shrinkage rate of the third sample was much faster than those of other samples. This may result from enhanced distribution and superplasticity of ultrafine particles. Also, the addition of titania affected grain size of alumina. The neck formation and growth in the third sample and the preheated sample have been investigated in situ using a heating TEM specimen holder. The neck formation in the preheated sample started at the lower temperature than that in the third sample. These experiments have revealed that the sintering rate is strongly dependent on processing and also the partial oxygen pressure.

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High-Resolution TEM Studies of Fullerenes. The extraction of macroscopic quantities of the solid phase of the carbon cluster C_{60} by the technique of Krätschmer-Huffman (KH) has stimulated a large number of investigations into their physical properties. Recently, further developments have led to the extraction and subsequent mass-spectroscopic characterization of larger clusters C_n, n = 100 to 330, termed giant fullerenes, from KH-carbon The paper presented here applies the technique of HRTEM to an investigation of the structure of the C_{60}, C_{70}, and giant fullerenes, including their transformation under electron beam irradiation. The results show significant differences in the structural response to electron beam irradiation among fullerenes of different sizes. The specimens were examined at 200 keV with a Hitachi H-8100 TEM.

An HRTEM image of a C_{60} thin film reveals the close-packed configuration of the f.c.c. structure viewed along the <111> and <110> orientations. There is strong evidence of electron beam damage. The lattice image of the initial crystal is transformed to one typical of an amorphous phase in 4 minutes. This change is consistent with the results of the electron diffraction patterns which is observed to change from a spot to a ring pattern. The lattice image of a C_{70} crystal reveals that the perfect lattices and the faults occur in clusters rather than being uniformly distributed throughout the grains, indicating the existence of short-range order in the C_{70} crystal lattices. Like the crystals of C_{60}, the crystalline phase of C_{70} is transformed into the amorphous phase by electron beam irradiation. The micrograph of giant fullerenes shows the circular features which are characteristic fringes of graphite with the typical layer spacing of 3.4 Å. We speculate that the graphitic features formed as a result of electron beam modification of the giant fullerenes. This result is in significant contrast to the results obtained on C_{60} and C_{70} in which the electron beam transforms the initial crystals into an apparently amorphous phase. The fact that the giant fullerene has more hexagons per molecule than do C_{60} and C_{70} may explain why the giant fullerenes are converted, upon irradiation with the electron beam, to a graphitic phase rather than an amorphous phase. The fact that the structures of the fullerenes are apparently modified by electron beams, and that the result depends on the molecule size, may eventually lead to a means for alteration and tailoring of fullerene molecules.

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Physico-chemical degradation of gold ion-exchange resins in hypochlorite solutions

During the past decade, the development of ion-exchange technology for the recovery of gold from hydrometallurgical process streams has advanced rapidly. Despite many attractive features of this technology, considerable resistance to the commercial implementation of ion-exchange processes remains, due in part to the many unknown features of the long-term operating characteristics of commercial ion-exchange resins. Conventional gold recovery is via cyanidation; for the treatment of refractory, carbonaceous gold ores, however, the use of halide ions, particularly chloride, for gold complexation is receiving increasing attention.

Two promising anion-exchange resins for the recovery of gold from chloride solution are SR-3 and PAZ-8. This study examines some physico-chemical properties of these resins when subjected to experimental conditions similar to those of a typical alkaline-chlorination gold recovery process. The extent of resin degradation was monitored as a function of reaction time, agitation and sodium hypochlorite concentration. Resin attrition and physical deterioration were observed using scanning electron-microscopy, while chemical decomposition and oxidation of both the polymer matrix and active functional groups were characterized by Fourier-Transform infrared spectroscopy using the diffuse-reflectance technique. It is shown that severe resin degradation, of both a mechanical and chemical nature, occurs on prolonged, agitated contact of the SR-3 resin beads with hypochlorite solutions at ambient temperature; the PAZ-8 resin is found to be more resistant to attack under these conditions.

BALDWIN, A.L. and WILSON, L.M. Department of Physiology, College of Medicine, University of Arizona, Tucson, AZ 85724. EDRF Affects Aortic Hydraulic Conductance. The endothelium is a physical barrier to fluid motion through the arterial wall. In this study, removal of endothelium increased arterial hydraulic conductance (L_p) at transmural pressures from 75 mmHg to 150 mmHg. At 50 mmHg, however, L_p was not changed by de-endothelialization. This suggests that the effect of the endothelium on L_p is not purely physical, and that at low pressures the endothelium produces a substance that increases L_p. To test whether EDRF acts in this way, the aorta in 8 rabbits was cannulated in situ and perfused at 50 mmHg with buffered saline containing 4% albumin. The output cannula was occluded and external diameter and transmural flow, hence L_p, were measured under 3 conditions: control; perfused with 10^{-4}M L-NAME, an inhibitor of EDRF synthesis; control. Inhibition of EDRF significantly reduced L_p from 6.79 ± 2.19 (SD) x 10^{-7} cm/s/mmHg to 2.59 ± 1.41 x 10^{-7} ($p<0.05$, paired Student t-test for all comparisons), but did not measurably alter vessel diameter. After removal of L-NAME, L_p returned to its initial control value. These results indicate that release of EDRF, and hence reduction of vascular tone, increases aortic L_p at 50 mmHg. This effect is probably mediated by alterations in arterial morphology. Preliminary ultrastructural investigations, using electron microscopy, suggest that removal of EDRF from the vessel wall causes disorganization of medial collagen fibers.

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An improved ultra rapid freezing system for metal cryofixation. The past decade has seen a decreasing interest in electron microscopy for classical morphological studies and an increasing interest in molecular biology, immunological procedures and elemental(ion) analysis. More scientists are turning to cryofixation as a method of preserving tissue in as life-like state as possible. Several methods of cryofixation may be used such as: Immersion(usually with fixed tissue) into liquid nitrogen or other cryogens such as halocarbons, propane or ethane. High pressure freezing, or Metal mirror cryofixation with either bench-bound or hand-held instruments. Metal mirror cryo fixation devices are popular because they give superior results, are less costly with respect to capital investment and pose no hazard or explosion risks. Although gravity and pneumatically activated table-top instruments provide reproducible results of high quality, they require the scientist to carry tissue to the freezer. Hand held versions allow the scientist to more closely freeze in-situ but have lacked the ability to control freezing variables. A pneumatically operated hand-held instrument is able to freeze both in-situ and in-vitro providing reproducible high quality freeze fixation as seen previously only with table-top units. The availability of such an instrument clearly improves the accuracy of many investigations. Lung tissue fixed in-situ while in the inflated state retains the pleural fluid layer which has never been shown this clearly before. Biological research, materials science and clinical pathology applications are currently under study at several major institutions.

CHANDLER, G. and KILLICK, D. Department of Materials Science and Engineering, University of Arizona, Tucson, AZ, 85721. SEM/EDX study of metallurgical crucibles from Niger, West Africa, CA. 500-1500 A.D. Trans-Saharan trade in slaves, gold, cloth, copper, and luxury goods was a major feature of West African history and is well described in documents from the eighth century A.D. on. The origin of this trade is unclear, and until recently most historians assumed that trans-Saharan trade only began in the Islamic era (i.e. eighth century A.D.). The site of Marandet, at the southern fringe of the Sahara, has abundant evidence (crucibles) of metallurgical activity in a region where such remains are otherwise rare. Radiocarbon dates of two crucible dumps excavated by Henri Lhote and three by Danilo Grebenart range from 550 ± 100 A.D. to 1420 ± 80 A.D., indicating that metallurgical activity at the site spans the beginning of the Islamic era south of the Sahara. We examined crucible specimens sent to us by Danilo Grebenart to investigate the possibility of import of the Marandet metals. Criteria used to distinguish local from imported metals include high arsenic content of local native-copper ores and the lack of any evidence of brass production south of the Sahara. BSE compositional images were employed to record alloy microstructures and clearly distinguish the metals and metal-rich glasses from the crucible material and products. EDX analysis of glass reaction products from crucible material in contact with the molten metals, and of the metals themselves, provide evidence that the metals are not of local origin. The composition of one glass strongly suggests that the metal melted in this crucible was a leaded brass, and was therefore an import from north of the Sahara. Future work (to include Pb isotope and microprobe) will compare Marandet metals to well-characterized North African alloys of the same age (e.g. Carthage).