placed in the tube furnace, wherein it was heated to 1,150 °C and held at this temperature for one hour. The test fixture was then retracted from the furnace (see Figure 1) and allowed to cool to 150 °C. The cooling process took approximately 5 to 6 minutes. Upon reaching 150 °C, the test fixture with the coupon was placed back in the furnace and reheated to 1,150 °C. The entire heating-and-cooling sequence was considered one cycle, and the lifetimes of the coupons were assessed on the basis of the numbers of cycles to failure.

The heat treatment of the NiCoCrAlY bond coats at reduced oxygen partial pressure yielded a significant increase in lifetimes: Coupons heat-treated to 1,750 °F (954 °C) at reduced oxygen partial pressure exhibited more than double the cycle lives of those containing as-sprayed NiCoCrAlY. This considerable increase in life can be attributed to the fact that selective oxidation of the aluminum and chromium in the bond coat yielded a graded interface. The heat treatment of the NiCrAlY bond coats yielded little or no increase in lifetimes.

The failure mechanisms of the coupons containing NiCoCrAlY bond coats differed from those of the coupons containing the NiCrAlY bond coats: The NiCoCrAlY-bond-coated specimens failed by decohesion and/or delamination at the interfaces between the top and bond coats. The NiCrAlY-bond-coated specimens underwent cohesive failure within the bond coats. Evidence of failure by these mechanisms can be seen in the left and the middle part, respectively, of Figure 2.

In an effort to reduce the extent of internal oxidation in the bond coats, platinum and rhodium barriers were employed as diffusion barriers. Initially, as-sprayed NiCoCrAlY-bond-coated coupons were coated with platinum to a thickness of 2 µm by physical vapor deposition (PVD). An example of a platinum diffusion barrier can be seen in the right part of Figure 2. The platinum-coated Inconel coupons were heat-treated to 1,800 °F (982 °C), then magnesium aluminate spinel top coats were thermally sprayed over the platinum coats. Rhodium diffusion barriers were applied to the surfaces of NiCoCrAlY-bond-coated coupons by pen electroplating. (Pen electroplating was investigated as a means of forming diffusion barriers because it is easy to perform and does not entail costly capital investment.)

The rhodium diffusion barriers yielded only a marginal increase in the lives of NiCoCrAlY-bond-coated coupons. However, platinum diffusion barriers applied by PVD in conjunction with reduced-oxygen-partial-pressure heat treatment yielded substantial increases in lifetimes. The platinum films were thick enough to constitute oxygen-diffusion barriers that slowed the growth of internal oxides by promoting the formation of alumina-rich scale at the interfaces between the top and bond coats. The best results achieved to date were realized by use of sputtered platinum diffusion barriers in conjunction with heat treatments to 1,800 °F (982 °C) at reduced oxygen partial pressures. This combination yielded a four-fold increase in the fatigue lives of NiCoCrAlY-bond-coated coupons.

Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Innovative Partnerships Office, Attn: Steve Fedor, Mail Stop 4–8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-17530-1.
MWCNTs Were Substantially Aligned along the flow direction after extrusion, as shown in this high-resolution scanning electron micrograph.

Multilayer Impregnated Fibrous Thermal Insulation Tiles

Temperature rises are limited by transpiration cooling.

Ames Research Center, Moffett Field, California

The term “secondary polymer layered impregnated tile” (“SPLIT”) denotes a type of ablative composite-material thermal-insulation tiles having engineered, spatially non-uniform compositions. The term “secondary” refers to the fact that each tile contains at least two polymer layers wherein endothermic reactions absorb considerable amounts of heat, thereby helping to prevent overheating of an underlying structure. These tiles were invented to afford lighter-weight alternatives to the reusable thermal-insulation materials heretofore variously used or considered for use in protecting the space shuttles and other spacecraft from intense atmospheric-entry heating. Tiles of this type could also be useful on Earth as relatively lightweight components of fire-retardant structures.

The SPLIT concept admits to so many different combinations of constituent materials, spatial distributions of the materials, and fabrication processes, that it is not possible to even list, much less summarize or describe all of them. Instead, a representative example must serve to illustrate the main principles. The starting material for fabricating a typical SPLIT is a porous substrate, having a void volume fraction of about 90 percent, that comprises a rigid tile or fabric made from any of a large variety of carbon fibers and/or ceramics fibers. The fiber composition can be the same throughout the thickness or can be graded: for example, it can differ among front, middle, and rear layers.

The front layer, which is the one to be exposed directly to intense heating, is typically impregnated with a thermosetting resin (e.g., a phenolic or a silicone). This layer becomes the first line of defense against intense heating: a large amount of heat is absorbed in the pyrolysis of the front polymer layer and is dissipated to the environment through a combination of outflow of the pyrolysis gas, and thermal radiation from the char layer formed in the pyrolysis. The outflow of the pyrolysis gas also provides further protection against heating by blocking the inflow of hot ambient gas.

The middle layer (if any) is typically not impregnated. The back layer is the one to be placed in contact or proximity to the structure to be protected. The back layer is initially impregnated with a thermoplastic polymer (the secondary...