This work examines the fabrication and thermal analysis of metal-carbon composite fibers prepared via an electrospinning process. The metal-carbon composite fibers of silver, copper, gold, and nickel were prepared by electrospinning of a composite solution of polyacrylonitrile (PAN) and metal precursor followed by heat treatment in air, nitrogen to 1000 degrees C and in 6% H2, respectively. Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), Fourier Transform Infrared Spectroscopy (FTIR), Energy dispersive spectroscopy (EDS) and Scanning thermal microscopy (SThM) were applied to characterize the metal-carbon fibers. TEM analysis showed a relatively uniform, contact-free distribution of the nanoparticles on the surface of the carbon fibers with size range of 3 nm-10 nm. Thermal analysis data showed an enhancement in the thermal conductivity of the nanomaterials when compared with the model PAN-based carbonized fibers. This was attributed to the incorporation of metal nanoparticles in the fiber matrix and on the surface.