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PREPARATION OF FIBERS WITH ENHANCED ULTRAVIOLET (UV) REFLECTANCE FOR ARCTIC CAMOUFLAGE

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Samples of fiber containing different loadings of Zirconium Oxide and Titanium Dioxide were prepared using standard melt spinning techniques. These samples were then compared to the Arctic Camouflage fabric using Ultraviolet (UV) photography. This report evaluates the parameters for incorporation of Zirconium Oxide into the fiber structure, including stability, processing parameters and the effect on the UV reflectance. The fiber containing Zirconium Oxide shows a higher UV reflectance than either the Arctic Camouflage Fabric or samples containing Titanium Dioxide.

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TABLE OF CONTENTS

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LIST OF ILLUSTRATIONS

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PREFACE

This report outlines a study to determine the processing and fiber forming characteristics of Nylon 6,6 fiber containing up to 10% loadings of Zirconium Oxide (ZrO_2) and Titanium Dioxide (TiO_2) for use in arctic camouflage. The evaluation was performed by the Fiber Project in conjunction with the Countersurveillance Section, Countersurveillance and Process Technology Branch, Materials Research and Engineering Division, Individual Protection Directorate.

The authors would like to express their appreciation to Ms. Margaret Goode, Individual Protection Directorate, for performing the Scanning Electron Microscope work and Mr. Thomas E. Rohnstock for conducting all of the Rheology work.

PREPARATION OF FIBERS WITH ENHANCED ULTRAVIOLET (UV)
REFLECTANCE FOR ARCTIC CAMOUFLAGE

1. INTRODUCTION.

Ultraviolet photography is a form of detection used in snow-covered environments. Snow has high UV reflectance values, whereas man-made objects generally have low UV reflectances. In fabric systems, especially, the use of Titanium Dioxide (TiO_2) as a dulling agent causes very low UV reflectance. For comparison, Figures 1 and 2 show a piece of the current arctic camouflage fabric against a snow background. Figure 1 shows the daylight appearance and Figure 2 shows the appearance with a UV filter. In Figure 1, the fabric is practically indistinguishable from the snow, and in Figure 2 it is readily discernible.

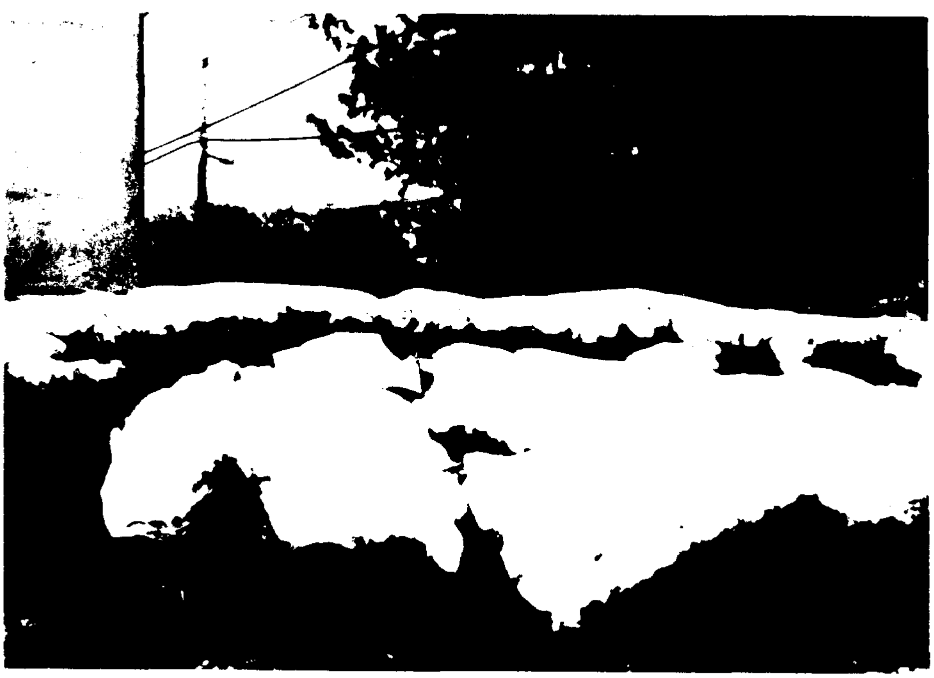

Figure 1. Comparison of the Arctic Camouflage Fabric to Snow in Full Sunlight.

Figure 2. Comparison of the Arctic Camouflage Fabric to Snow using a UV Filter.

Currently, vehicle paints for use in arctic areas contain Zirconium Oxide (ZrO_2), which has reflectance values in the UV that provide a good match to snow.[1] Attempts have been made to treat fabrics with coatings containing ZrO_2 for use in camouflage nets.[2,3] The coating increases the fabric weight significantly which would be unacceptable if applied to military uniform fabric.

Substituting ZrO_2 for TiO_2 and adding to the solution prior to extruding fiber, in place of topically treating the fabric, would increase UV reflectance without adding weight. In an effort to add UV protection to the individual soldier's uniform without adding excess weight, a preliminary study was undertaken to investigate methods of incorporating ZrO_2 into nylon fiber.

The study was subdivided into four tasks:

a. Determine the particle size distribution for ZrO_2.

b. Evaluate processing characteristics and degradation.

c. Prepare fibers containing up to 10% of ZrO_2 and TiO_2 in Nylon.

d. Compare UV reflectance of fibers to Arctic Camouflage Fabric.

Each of these tasks is addressed in more detail in the following sections.

2. PROCEDURES.

a. Particle Size Analysis:

Preparation of continuous filament textile grade yarns containing inorganic pigments or additives requires evaluation of the particle size to prevent clogging of the spinnerette orifice and formation of weak points in the yarn. A textile grade filament may have diameters less than 0.02 mm (20 microns) so particle sizes for the pigments used need to be much less than this diameter. As an example, TiO_2 can be obtained with uniform particle sizes of 0.8 microns which are easily dispersed and do not clog the orifice.[4] The spinnerette orifice used for this work was 0.25 mm. or 250 microns. The target diameter for the filaments was 20-25 microns.

Figure 3 is a Scanning Electron Micrograph (SEM) of a sample of ZrO_2 at a magnification of 1500X. The instrument used for this micrograph was an AMRAY Model 1000A Scanning Electron Microscope.

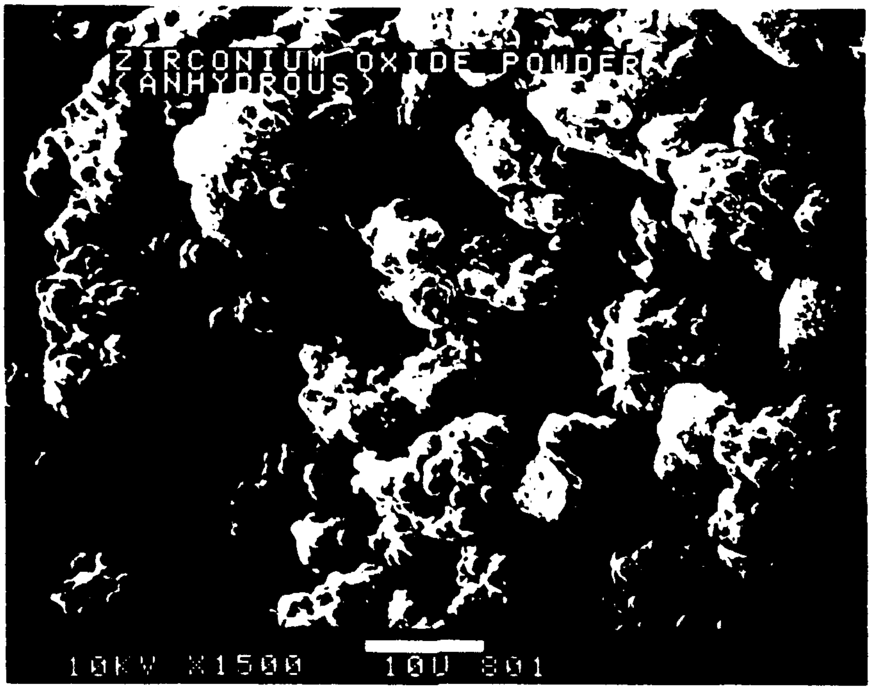

Figure 3. Scanning Electron Micrograph of Zirconium Dioxide.

The ZrO_2 used consisted of individual particle sizes between 2 and 3 microns as determined by randomly choosing three areas and measuring all particles within the areas. This data is listed in Table 1. Agglomeration (clusters) of particles were as large as 20 microns. However, these break up during the extrusion process or are filtered out by a series of fine screens (50-300 mesh) located before the spinnerette. Particle size was not evaluated for the TiO_2 since it was already in concentrate form and had been used successfully for fiber spinning on previous occasions. No anti-agglomerates were evaluated during this work.

Table 1. Particle and Cluster Size Analysis.

Particle Measurements (microns)			
	Area #1	Area #2	Area #3
Range	1.56 - 2.68	1.67 - 4.03	2.01 - 3.69
Average	2.15	2.40	2.81
Cluster Measurements (microns)			
	Area #1	Area #2	Area #3
Range	6.04 - 19.26	9.07 - 19.14	5.71 - 12.09
Average	6.36	13.54	9.07

b. Polymer Stability:

Prior to attempting fiber preparation, samples of Nylon 6,6 containing 5% of ZrO_2 and 5% TiO_2 were tested for stability and flow characteristics using a Haake Buchler (System 40) Torque Rheometer. Testing was conducted with the 600 Series Mixer set at a constant temperature profile of 295^{o}C. With rotors turning at a constant speed of 20 RPM, a sample charge of 51.3 grams was added to the mixer. Figures 4 and 5 show torque values vs. time for the samples containing 5% ZrO_2 and 5% TiO_2 in nylon 6,6 resin, respectively.

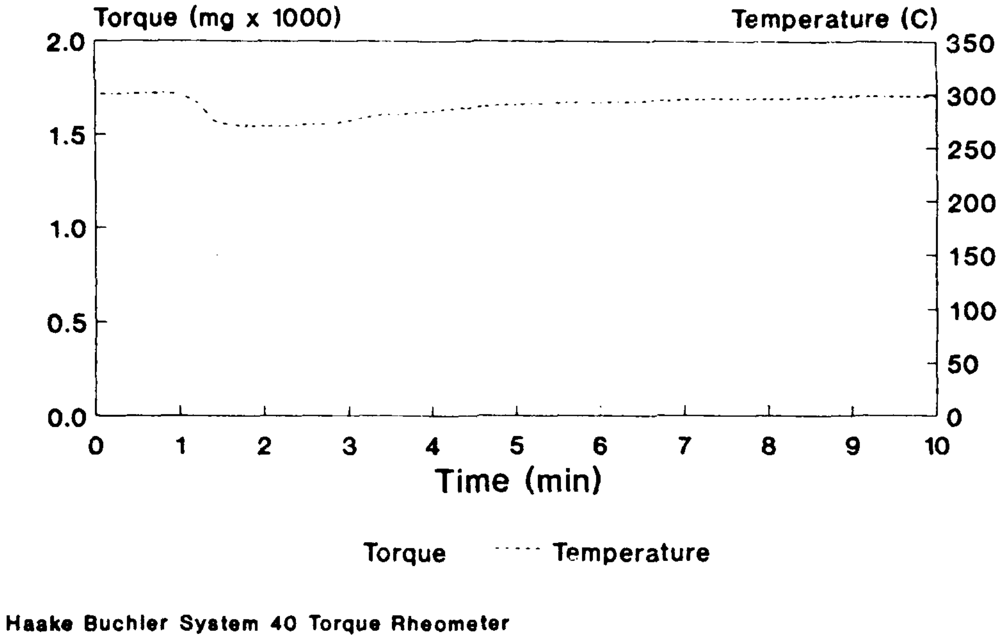

Figure 4. Torque vs. Time Curve for Nylon 6,6 containing 5% Zirconium Oxide.

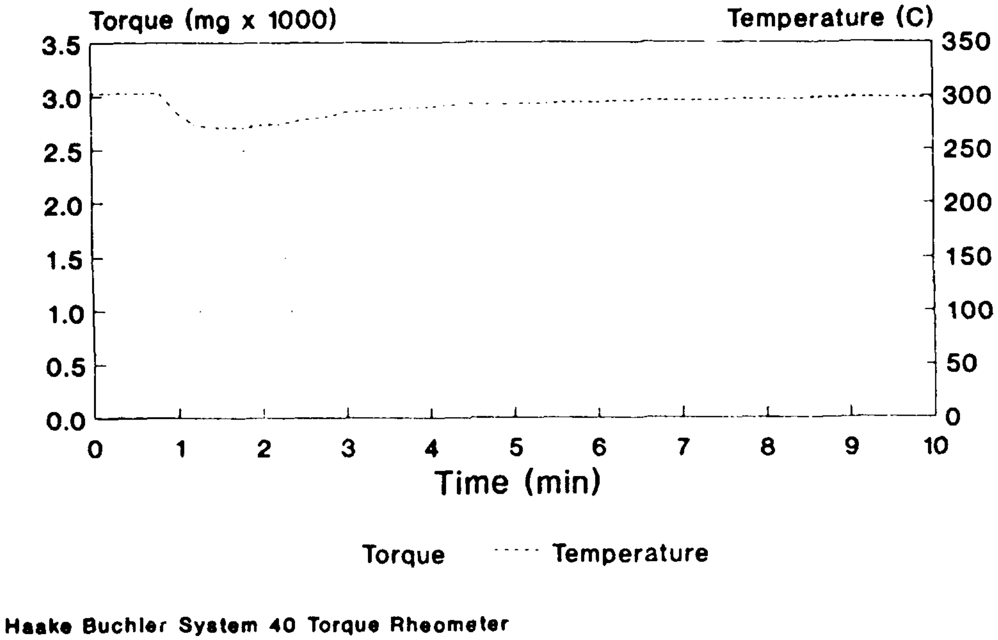

Figure 5. Torque vs. Time Curve for Nylon 6,6 containing 5% Titanium Dioxide.

The torque curves for all of the samples exhibit standard behavior. The curves show a sudden increase in torque, which is known as the loading peak, with a simultaneous reduction in temperature as the cooler material takes on heat from the mixer. As the material melts and becomes more fluid, a substantial reduction in torque is seen with a corresponding stabilization of temperature. In both Figures 4 and 5, a shoulder is apparent on the loading peak. This was caused by a small amount of material that caught in the loading chute and fell into the mixer after most of the sample had already melted. This does not indicate any substantial variation from standard behavior. A standard torque curve for nylon 6,6 is included as Figure 6.

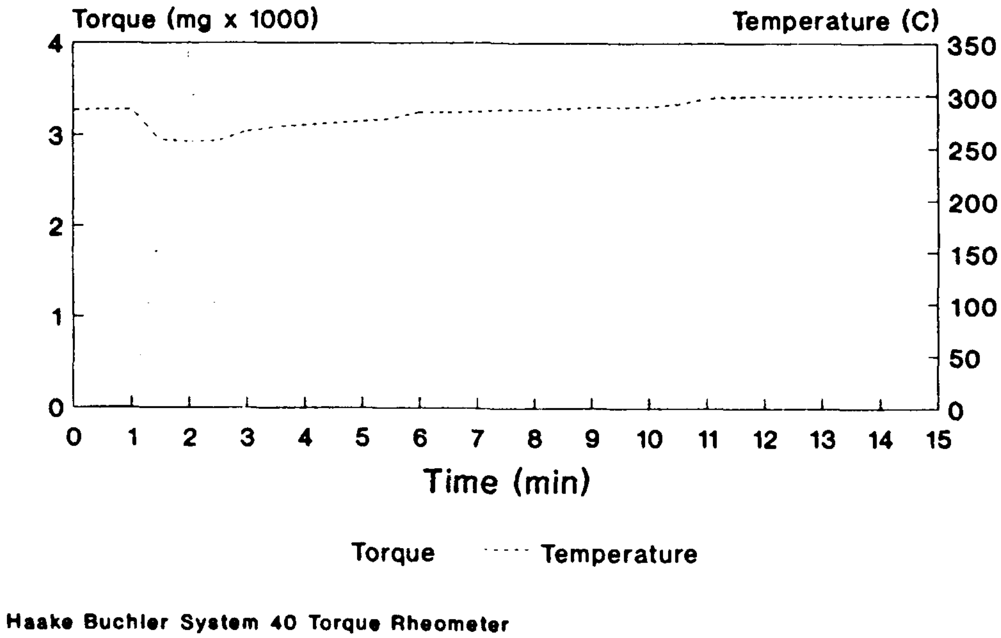

Figure 6. Torque vs. Time Curve for Nylon 6,6 Standard.

From this data, it can be projected that there should be no major problems in processing due to instability. All indications are that the samples will process at conditions similar to those for nylon 6,6 alone.

c. Fiber Preparation:

Fiber spinning was performed using a Research Spinning Machine (REX) designed by Hills Research and Development Inc. Spinning conditions are listed in Table 2.

Table 2. Research Spinning Machine Parameters for Fiber Preparation.

Extruder Temperatures

Zone 1	-	270°C
Zone 2	-	275°C
Zone 3	-	285°C
Spin Head	-	290°C

Extruder Pressure	-	200 psi.
Pack Pressure	-	900 - 1000 psi.
Draw Ratio	-	2.5 : 1

Using the extrusion conditions outlined in Table 2, samples were prepared containing 5 and 10% ZrO_2. In addition, a sample of fiber containing 10% TiO_2 was also prepared. Additional levels were not attempted due to limited quantities of material. The only difference between the processing characteristics of the two materials was the tendency for the pack pressure to be slightly higher when using ZrO_2.

Fiber samples were not drawn down to the full extent possible due to the limited amount of material and small samples attempted. Substantial run times are required to optimize the throughput and draw ratio to obtain a textile grade fiber. Drawing a fiber introduces a higher degree of orientation (i.e. crystallinity) to the fiber and consequently more strength. Samples were wound onto sample cards and submitted for evaluation using ultraviolet photography.

All materials were dried at 125°C under vacuum (30 in. Hg.) overnight. The spinnerette used was a 48 hole jet with a round cross section. Sample descriptions are listed in the Appendix.

d. Ultraviolet Photography:

Photographs were taken of the samples using the procedure outlined in Kodak Publication M-27. A Kodak 18A filter, which transmits only long wave ultraviolet (from about 320 to 400 nm), was used to isolate the ultraviolet reflectance of the samples on film. A 35 mm camera with Kodak 400 speed film for color prints was used with varying exposure times.

The sample cards were placed next to a sample of the arctic camouflage material (MIL-C-3924G, Cloth, Oxford, Cotton Warp and Nylon filling, Quarpel Treated, Class 1) in full sunlight and UV photographs were taken. Figure 7 shows the samples containing 5 and 10% ZrO_2, and 10% TiO_2 with the arctic fabric, in full sunlight without the filter and Figure 8 identifies the sample position. The samples show no apparent differences.

Figure 9 shows the same arrangement as Figure 8 through a UV filter. The sample containing 10% TiO_2 appears very dark when compared to the fabric, while the 10% ZrO_2 is slightly brighter than the fabric, and the 5% ZrO_2 appears much brighter than the fabric. TiO_2 is typically used as a dulling agent at approximately a 2% loading, to eliminate the luster of nylon. This is the case with the nylon fill used in the arctic camouflage fabric. The loadings of 5 and 10% were used to exaggerate differences between the samples. Further spectral work will be required to more fully describe this effect, in addition to optimizing concentration levels for maximum reflectance at minimum concentration.

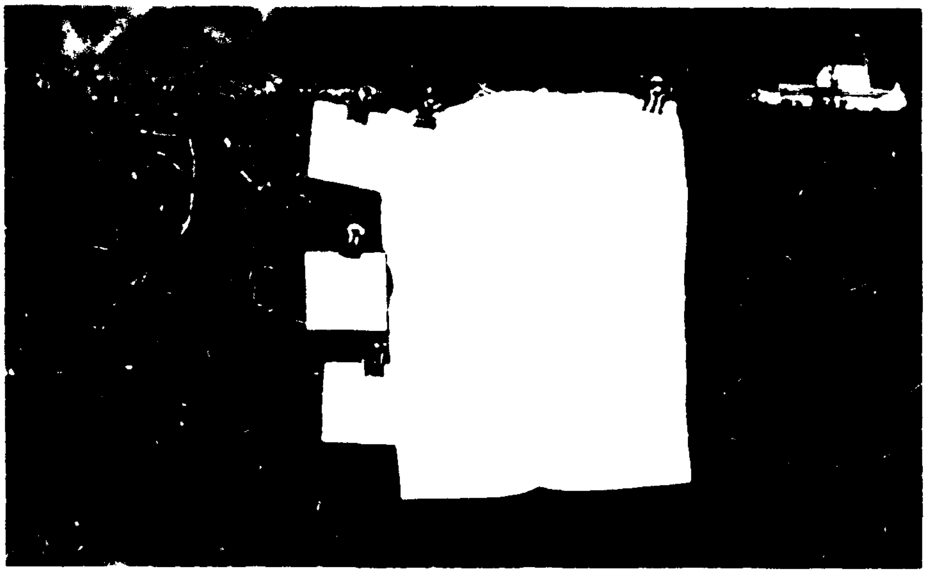

Figure 7. Comparison of Samples containing ZrO_2 and TiO_2 to Arctic Camouflage Fabric in Full Sunlight.

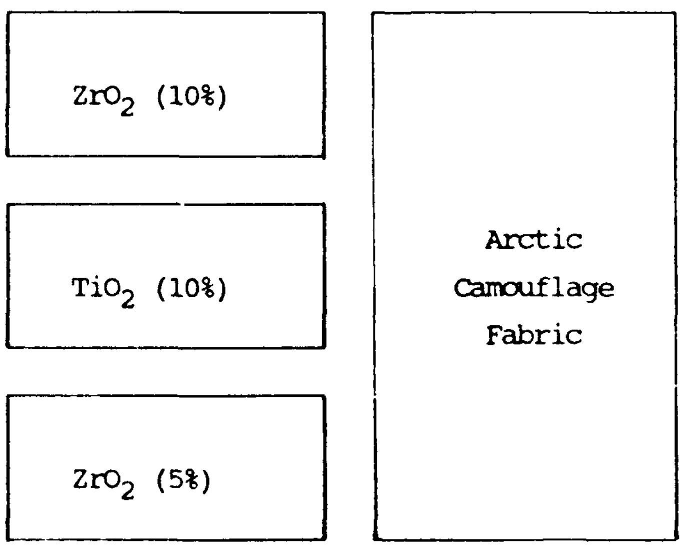

Figure 8. Sample Arrangement in Figure 7.

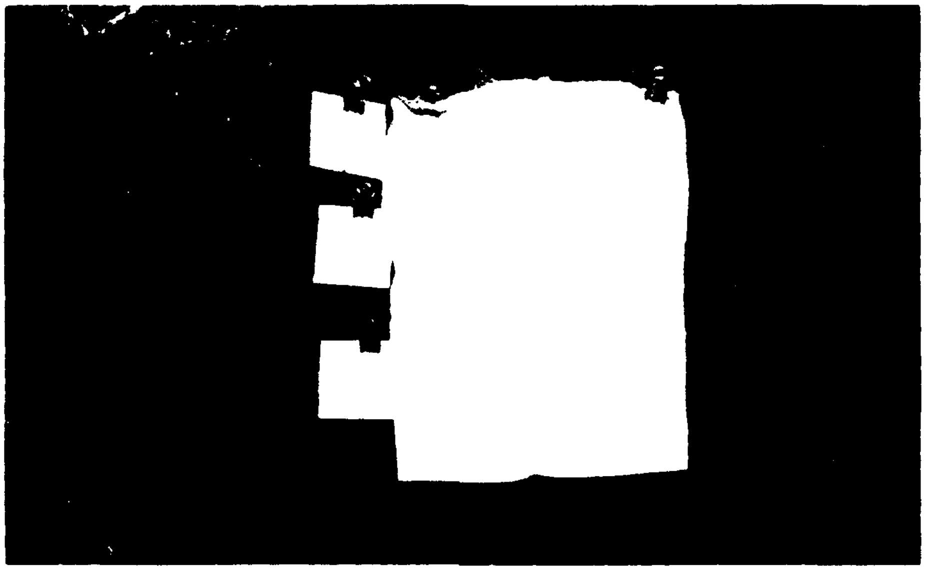

Figure 9. Comparison of Samples containing ZrO_2 and TiO_2 to the Arctic Camouflage Fabric in Full Sunlight using a UV Filter.

CONCLUSIONS AND RECOMMENDATIONS:

Preliminary evaluation of ZrO_2 as a potential substitute for TiO_2 in yarns used in the arctic camouflage material to increase UV reflectivity shows promise. Comparisons using UV photography show distinct differences between samples containing 5 or 10% ZrO_2 and a sample containing 10% TiO_2. Further research will be required to determine optimum concentration levels of ZrO_2 as compared to TiO_2, which is a standard additive to synthetic yarns. Additional process work will be required to prepare a textile grade yarn containing the optimized concentrations of ZrO_2 and a sample of fabric prepared from this yarn for comparison to the current arctic camouflage fabric.

This document reports research undertaken at the U.S. Army Natick Research, Development and Engineering Center and has been assigned No. NATICK/TR-92/014 in the series of reports approved for publication.

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3. Desert and Arctic Camouflage Garnishes, Gordon H. Johnson and Robert W. Pelz, Ferro Corporation, Independance, Ohio, report for Belvoir, AD B009230, Oct. 1975.

4. Man-Made Fibres, R.W. Moncrieff, Sixth Edition, Newnes-Butterworths, 1975.

APPENDIX

APPENDIX

SAMPLE DESCRIPTION:

Zirconium Oxide (ZrO_2)
Purified (Anhydrous)
Fisher Scientific Lot No: 897129
2 micron particle size

Titanium Dioxide (TiO_2)
Imperial Pigment Colors
50% Titanium White (Concentrate)
Hercules Incorporated, Batch 6-01876

Zytel Nylon 6,6 Resin
E.I. Dupont DeNemours & Co. Inc.
Lot No. VJ44C57

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