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REVIEW OF INFRARED HEATING AND ITS APPLICATION TO UHMWPE

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CONTENTS

Abstract

The purpose of the cooperative research and development agreement between Smith & Nephew, Inc. (S&N) and ORNL was to explore the effects of infrared radiation heating on the physical properties of UHMWPE. Certified bars of ram extruded GUR 1050 UHMWPE were exposed to infrared heat under various conditions at ORNL. For comparison, specimens from the same bar stock were remelted using a conventional oven. Specimens from all conditions were tensile, impact, and solvent extraction tested at S&N for comparison to unmelted control specimens. Although heating conditions were not optimized, infrared radiation was found to be extremely efficient at heating UHMWPE, melting bar stock up to five times faster than a conventional oven without degrading the bulk of the material. Key physical properties such as percent elongation and impact strength increased after remelting, indicating that the degree of chain interpenetration and entanglement was augmented. This increase in consolidation was supported by the results of boiling xylene extraction tests, which showed that the percentage of soluble components and the swell radio were significantly lower for the remelted UHMWPE specimens than for the controls. The solvent extraction testing also indicated that infrared remelting might have induced cross-linking in the UHMWPE bar stock. Significant decreases in yield strength and tensile elastic modulus that were observed after remelting were not expected to be problematic because the yield strengths remained above ASTM standards and the tensile elastic moduli of most treated specimens were within the range specified in the FDA guidance document for UHMWPE.

CRADA Benefits to DOE

The benefit to DOE was that knowledge was gained in low-temperature, vacuum heat-treating of polymers.

Technical Discussion

Henry Ford, the father of the efficient production line, was the first to use simple infrared on a large scale back in the 1930s, when he introduced ovens to bake new resin finishes onto cars by the optical projection of radiant energy from electrical incandescent lamps. The radiant heat is absorbed immediately, allowing rapid curing, thus, eliminating the need for forced ventilation. Preheat times were considerably reduced [1]. Today, infrared technology can be found in use across a wide range of industrial applications. It is used in the electronics industry for drying solder resists, in food processing for browning and sterilization, in finishing for stoving and curing, in textiles for drying and sealing, in plastics for softening, in printing for drying, and in engineering for preheating and shrink fitting. As is evident in the above list of applications, infrared conventionally has been used for low temperature non-robust applications. Advances in lamp design and furnace designs have enabled heat fluxes as high as 236 W per linear centimeter to be delivered to a part utilizing tungsten halogen based lamps on a commercial scale. Continuous heat flux furnaces have been developed that can readily deliver 43 W per square centimeter. These furnaces have been shown to be robust enough to heat treat and temper unidirectionally a 682-kg die block with a 88-kW infrared furnace and austenitize one ton of steel per hour for a 300-kW circular infrared furnace.

Infrared radiation is energy transported by electromagnetic waves propagating at the speed of light. The absorption of the infrared waves by a material results in a transition to thermal energy and subsequent heating. Of the three heat transfer mechanisms (i.e., conduction, convection, and radiation), radiation, and particularly infrared radiation, has some unique advantages. Infrared heating provides: (1) an inherently clean non-contact heating method; (2) high heat fluxes resulting in heating rates up to 200°C/s (materials dependent) due to the fact that only the sample is heated to the desired temperature; (3) provides heating only to the part (i.e. low thermal mass); (4) fast cooling rates due to the "cold wall" nature of the process in which only the sample is heated; (5) high heat fluxes, which can be delivered unidirectionally over large areas; and (6) a precise process, which is essentially shape independent, utilizing proper furnace designs.

When determining the potential uses of infrared, the following areas are important:

1. Absorption characteristics of the material being heated.
2. Power density of the radiating area on the part.
3. Ratio of convected heat to radiant heat.
4. Geometry of infrared emitters and reflectors (including furnace design).
5. Type of control required.

To better understand the applicability of infrared, the factors involved with the transfer of radiant heat will briefly be discussed.

All bodies radiate energy as a function of their absolute temperature, as defined by Stefan-Boltzmann Law:

$Q = kT^4$,
Q = total emissive power (watts/cm^2),
k = Stefan-Boltzmann constant = 5.56×10^{-12} (watts/cm^2-°C^4),
T = absolute temperature (°C). (1)

Infrared energy is the portion of the electromagnetic spectrum between 0.78 and 1000 μm. The infrared electromagnetic spectrum can be divided into three divisions: (1) short wave 0.78 to 2.0 μm, (2) medium wave 2.0 to 5.0 μm, and (3) long wave 5.0 μm to 1 mm. The actual emission spectrum of a given source is dependent upon its temperature. Increasing the source temperature will result in shorter overall wavelengths of the energy. This also corresponds to an increase in the overall emissive power per the equation above [2].

In order to understand which parameters are important in rapid infrared heating, one can consider the general equation for the heat transfer between the source and target:

$$Q = (FV) \times (ES) \times (AT) \times (k) \times (TS^4 - TT^4)\,, \qquad (2)$$

where

Q = heat transfer between the source and target (W/cm^2),
FV = view factor between the source and target,
ES = emissivity factor of the source,
AT = absorption factor of the target,
k = Stefan-Boltzmann constant,
TS = absolute temperature of the source, and
TT = absolute temperature of the target.

The view factor term is the fraction between 0 and 1 that quantifies the amount of radiant energy emitted from the source that fall incident upon the target. Control of the heating rate is accomplished through varying the source temperature. The absorbed heat transfer (Q) results in a temperature rise of the target as defined by:

$$T = \frac{(Q)\times(A)\times(t)}{(M)\cdot(Cp)}, \tag{3}$$

where

T = product temperature rise (K),
A = area of target (cm^2),
t = heating dwell time,
M = mass of the target (kg), and
Cp = target specific heat (W-s/kg-K).

Increased temperature rise can be achieved by increasing the heat transfer, dwell time, or the amount of incident infrared on the target. The wavelength of light utilized in the ORNL experimental system, which is approximately 1.2 µm spectrum, allowed for maximum percent emissive power. Spectral distribution of radiant energy follows Plank's Law which, as a function of temperature and the wavelength considered, gives the monochromatic emissivity $M\lambda$ of a black body for wavelength l [3-5]:

$$M\lambda = C_1/((\lambda^5(e^{C_2/\lambda T} -1)), \tag{4}$$

where

$M\lambda$ = radiant power in watts per square micron, corresponding to wavelength λ,
C_1 = constant is 3.741×10^8 W × µm^4/m^2,
C_2 = constant is 14,388 µm × K;
λ = wavelength in microns;
T = temperature of the filament, K.

Emission of radiation, however, is not emission of heat. It is only when a body absorbs radiation that it is converted into heat. The overall absorption capability, α, at a point is the ratio between the flux absorbed and the incident flux. For all known bodies, this ratio is less than one, since part of the radiation is reflected and, if the body is not opaque, part of the radiation is transmitted. If ρ is the reflection factor or reflectivity and τ the transmission factor or transmissivity:

$$\rho + \tau + \alpha = 1 . \tag{5}$$

The maximum monochromatic emissivity, or emissive power referenced to wavelength, at absolute temperature of the filament corresponds to the wavelength λ_μ, as determined by Wein's Law:

$$\lambda_\mu \times T = C \tag{6}$$

where

λ_μ = wavelength in microns,
T = absolute temperature in degrees Kelvin, and
C = constant equal to 2,898.

Wein's Law is only an approximation and can only be applied for wavelengths between 0.3 and 10 μm, which is the range utilized in this work. Ninety-five percent of the emitted energy is radiated within the limits of 0.5 and 5 μm. An emission of 1% falls below 0.5 μm and 4% above 5 μm. Therefore, as is intuitively evident, production of energy in the maximum percent emissive power range can be extremely efficient, 95%.

Infrared radiation for industrial applications is typically produced by quartz-type radiators, which ensure high-power densities, maximum heat efficiency, flexible design parameters, and easy control [2]. The quartz-type radiators consist of a tungsten filament encapsulated in a quartz envelope in which halogen is mixed with an inert atmosphere. As previously discussed, the tungsten is heated as high as 2977°C, which results in shorter overall wavelength of energy and increased emissive power. To prevent filament evaporation which could cause blackening of the tube and diminish its energy efficiency and service life, a halogen (generally iodine) is added to the inert gas filling the tube. The evaporated tungsten reacts with the iodine to form tungsten iodide, which is volatile. This iodine is taken by convection to the filament, where at high temperature it decomposes and redeposites tungsten on the filament. This releases the iodine back into the atmosphere of the tube [1-5].

The lamps emit energy in the infrared region, which lies immediately above the visible spectrum. Lamp energy concentrated in the near infrared spectrum, with typical full voltage peak color temperature output at 2227°C (4041°F). For halogen cycle lamps, lamp energy is also concentrated in the near infrared spectrum, but the typical full voltage peak color temperature is 2977°C (5391°F). As can be seen from these figures, the primary wavelengths are between 0.3 and 5 μm and by varying operating voltage, the peak wavelength can be varied from 0.89 to 2.0 μm.

Another very important feature of infrared lamps is there relatively short heat-up and cool-down times. As can be seen, start-up and shutdown times on the order of 1 s are common.

A 100-kW infrared furnace consists of an array of tungsten halogen lamps mounted in a given geometry to direct the disperse infrared in a direction of choice. The furnace walls are polished and water-cooled to minimize the absorbed infrared and thus maximize the reflected. Infrared heating, in this case, is a cold-wall process in which only the sample is heated to the desired temperature. This allows for fast heating and cooling, precise control of the temperature, instantaneous starting and stopping, and low operating costs. The emissive power can be delivered effectively with over 90% efficiency to a wide variety of part sizes and geometry's. Joining applications such as advanced materials joining, titanium aluminide, TiAl, iron aluminide, titanium matrix composites, automotive parts, titanium to steel for armor applications, and others which take advantage of one or more of the salient features mentioned. Other applications include unidirectional heating of die blocks for rapid preheat, preferential tempering, predetermined hardening, and preheat for powder metallurgical sheet, steel, and wire. In the many cases mentioned, the infrared processing technique results in tremendous cost savings through reduced processing time, reduced operating cost, and environmental friendliness. The underpinning science and system survivability designs have been developed at ORNL in reference to infrared with these types of applications.

The purpose of this investigation was to determine the effects of remelting, particularly by infrared radiation heat treatments, on the physical properties of UHMWPE barstock. The postulate was that the infrared radiation would resonate carbon-carbon and carbon-hydrogen functional groups within polymer chains in much the same manner as infrared spectroscopy and, thus, facilitate chain movement, interpenetration, and entanglement. It was anticipated that the increased interpenetration and entanglement of polymer chains would result in better consolidation and physical properties than untreated controls. After exposing UHMWPE bar stock to infrared radiation under a variety of environmental conditions, the tensile properties and impact strength of the materials were measured and compared to conventional oven remelted and control specimens. Furthermore, solubility tests were conducted to determine if the infrared treatments degraded the polymer systems.

Materials and Equipment

The following materials and equipment were used in this investigation.

- 5.7-cm (2.25-in.) -diam UHMWPE bar stock
- Radial infrared heating system (see Figure 8) equipped as follows:
    - ➢ 10.2 cm (4 in.) diameter quartz chamber

  - ➢ 22 - 16.5 cm (6.5 in.) long, 200 W/linear in. tubular quartz tungsten halogen filaments arranged circumferentially around quartz chamber
  - ➢ Yokogawa controller
- LabView Data Aquisition System software - Version 5.0
- DXL high elongation extensometer (MTS Systems, Eden Prairie, MN)
- ASTM D 638 Type IV die (Dewes Gumbs Die Co., Inc., New York, NY)
- Tinius Olsen test frame (*)
- Impact tester (Testing Machines, Inc., Amityville, NY)
- Heating mantle
- 1000 mL glass round bottomed flask with ground glass joint (24/40)
- Condenser with 24/40 joint
- Vacuum oven
- Stainless steel wire and wire mesh
- Thermocouples
- Reagent grad xylene (Aldrich Chemicals)
- Di-t-butyl-4-methylphenol (Aldrich Chemicals)

Methods

Sample Preparation

Method Development and Remelting by Infrared Oven

Because this was a feasibility project with no previous experience with heating UHMWPE, the first portion of this study was spent determining the thermal response of the polymer to the infrared heat. Thermocouples were on the surface and, in some cases, in the core and at half the radius of 3 to 4" long bars of the 5.7 cm (2.25 in.) diameter UHMWPE. Several heating cycles were conducted using different intensities (i.e., percentage of rated voltage) while monitoring changes in temperature and appearance of the bar. After finding an appropriate infrared heating method that melted the UHMWPE bar stock while avoiding significant surface degradation, the 5.7 cm (2.25 in.) diameter bars that were approximately 6.15 cm (6 in.) long specimens were prepared for physical property testing.

Remelting by Conventional Oven

UHMWPE specimens of the same dimensions used in the infrared treatment were placed in a vacuum oven set at 150°C. The temperature was increased 160°C and a vacuum of 4.4 to 4.6 in Hg was applied. Complete melting was achieved after 5.5 h at which time the heat was turned off and nitrogen gas was introduced to bring the pressure in the oven up to atmospheric pressure. The bars were allowed to cool to room temperature before removing from the oven.

Tensile Testing

Sheets 3.2 ± 0.4 mm (0.13 ± 0.02 in) thick, at least 115 mm (4.5 in.) long, and at least 19 mm (0.75 in.) wide were machined from the heat treated specimens. The sheets were cut into ASTM D 638 Type IV tensile specimens using a die, which resulted in a trapezoidal cross-section. The dimensions of the gage section of each specimen were measured using calipers such that the cross sectional area could be calculated. On an individual basis, test specimens were placed in the mechanical grips of a Tinius Olsen test frame and the two clamps of the extensometer were attached one inch apart within the gage section of the specimen. A static tensile load was applied to the specimen at a displacement rate of 5.08 cm/min (2 in./min) per ASTM D 638. Load and strain were recorded directly from the test frame and the extensometer, respectively, using a PC equipped with LabView software. The data were translated into Excel spreadsheet format. The stress at each displacement was calculated as the applied load divided by the initial cross sectional area of the gage section. The ultimate tensile strength (UTS), yield strength (YS), percent elongation (% elong.), and Young's modulus (E) were determined for each specimen.

Impact Testing

Izod double notched impact test specimens were machined and tested at S&N in accordance with ASTM F 648. In summary, specimens were notched with a razor blade and impacted with a pendulum hammer. Impact strength was determined by dividing the energy loss of the pendulum during impact by the effective cross sectional area of the specimen.

Solution Testing

The method used to determine swell ratio and percent extractables was a modification of ASTM D 2765-95. Approximately 2.0 x 2.0 x 2.0 mm (0.08 x 0.08 x 0.08 in) specimens were fabricated from the control, oven remelted, and vacuum remelted UHMWPE bars. The specimens were weighed to the nearest 0.01 mg and recorded as the initial dry weight (W_o). Each specimen was placed in a stainless steel mesh basket, which was preweighed (W_b) to the nearest 0.01 mg, connected to a stainless steel wire, and submerged in boiling xylene for 24 hours. The xylene contained di-tert-butyl-4-methylphenol at 0.5 wt % to prevent degradation. After removing from xylene, baskets containing specimens were removed and placed on a piece of filter paper to extract excess xylene. The swollen specimen and the basket were then weighed together ($W_{bs} = W_b + W_s$) or separately if the specimen was intact. The specimen and basket were then placed in a 100 °C vacuum oven equipped with a cold trap. After approximately 24 hours, the specimen (and basket) was repeatedly weighed until equilibrium. The final weight of the dried specimen basket was recorded as $W_{bd} = W_b + W_d$.

Based on the research of Flory et al. [7], the following equations were used to calculate the swell ratio (SR), percent extract (%E), and average molecular weight between cross-links ($\overline{M}_c$), respectively:

$$SR = \left[\left(W_{bs} - W_{bd}\right)/\left(W_{bd} - W_{b}\right)\right]K + 1 \qquad (7)$$

$$\%E = \left[W_{o} - \left(W_{bd} - W_{b}\right)\right]/W_{o} \leftrightarrow 100\% \qquad (8)$$

$$\overline{M}_{c} = -V_{1}\rho\frac{\left(\Phi^{1/3} - \Phi/2\right)}{\left[\ln(1-\Phi) + \Phi + 0.3\Phi\right]} \qquad (9)$$

where

K = ratio of the density of polymer to that of the solvent (assumed as1.1),
V_1 = molar volume of the solvent (122 cm^3/mol),
ρ = polymer density (assumed to be 0.93 g/cm^3), and
Φ = the volume fraction of polymer in the swollen gel = $(SR)^{-1}$.

Statistical Methods

The data were analyzed using single factor analysis of variance (ANOVA). If significant differences were found ($p<0.05$), then comparisons between individual treatments and the control were made using a two-sample t-test assuming unequal variances. The confidence coefficient $(1-\alpha)$ was selected as 0.95 for the analysis.

Results

Infrared Heating Method Development

A typical heating profile for the surface of a 5.7 cm (2.25 in.) diam UHMWPE bar heated by 22 lamps at 11 to 16% rated voltage in an argon environment. As expected, the surface of the bar heated the most rapidly and was tracked by the one-quarter depth region and the core. The fact that the core was heated immediately upon exposure to infrared indicated that the radiation was achieving good penetrating even though the polymer was opaque. It was observed that prolonged exposure to high intensity infrared caused extensive degradation at the surface, i.e. the polymer began to flow. Consequently, the number of active filaments was reduced from 22 to 3. The bar stock specimens were found to melt in a relatively short amount of time using 3 filaments at 22 to 23% of their rated voltage. This general heating method was used to create specimens used for testing.

Tensile Testing

The stress-strain diagrams, tensile property data, and statistical comparisons are given in Appendix B. The statistical comparisons for the tensile tests are summarized in Table I. In general, it was observed that the percent elongation increased after remelting by both conventional oven and infrared heating and that modulus decreased after remelting.

Table I. Statistical comparisons between heat treatments and control.

Comparison **Control vs.**	**Property** **Significant difference? (Y/N - highest)**				
	UTS	**YS**	**% elong.**	**E**	**Impact**
Oven remelted	Y - oven (p =	Y - control (p = 1.99E-	Y - oven (p = 0.001)	Y - control (p = 3.19E-	Y - oven (p = 1.65E-
Infrared/argon remelted	N	Y - control (p = 0.0028)	Y - IR/argon (p = 3.54E-4)	Y - control (p = 8.28E-6)	N/A
Infrared/vacuum remelted - 1	Y - IR/vac. (p = 0.026)	N	Y - IR/vac. (p = 1.59E-5)	Y - control (p = 1.24E-6)	N/A
Infrared/vacuum remelted - 2	N	Y - control (p = 2.41E-4)	Y - IR/vac. (p = 4.4E-4)	Y - control (p = 1.12E-4)	Y - IR/vac. (p = 3.3E-4)

Impact Testing

It was observed that remelting the control specimen via conventional oven or infrared oven increased the impact strength by about 14%. No differences were found between the oven and infrared remelted specimens, however, the data from the infrared treatment had a higher variability. To determine if the variation in impact strength was caused by the relative position of the specimens within the UHMWPE bar to the infrared lamps, the average impact strengths of specimens taken from different locations within the treated bar were plotted. It was observed that the specimens that were closer to the core of the bar had higher impact strengths.

Solution Testing

The raw data, calculated swell ratio and percent extractables and comparative statistics for the control, oven remelted, and infrared-vacuum remelted specimens. The comparisons between swell ratio and percent extractables were calculated. Both of the remelting treatments decreased the swell ratio and percent extractables significantly in comparison to the control. These values tended to be lower for the infrared treatment but the differences were insignificant.

Discussion

As part of this investigation, UHMWPE bar stock was remelted using an infrared radiation and conventional oven to determine if there were any unique advantages to infrared heating. Infrared radiation heating was found to be much more efficient, completely remelting the 5.7-cm (2.25-in.) -diam UHMWPE bars approximately four to five times faster than a conventional oven set at 160°C and under vacuum. Additionally, it was observed that the core of UHMWPE bars heated very rapidly due to effective penetration of the intense infrared radiation. Unfortunately, the emission of the tungsten filaments on the infrared oven used in the investigation was too high for polymers. The surface of the bars underwent thermal degradation, had reduced viscosity, and began to flow when all of the filaments were used, even at a very low percent rated voltage. Thus, only 3 of the 20 filaments could be used at 20 to 25% of their rated voltage to melt the UHMWPE. Using only three filaments decreased the homogeneity and penetration of the radiation and, as mentioned before, the relatively low power output by each filament produced relatively large wavelengths that did not penetrate as effectively. The changes in impact strength as a function of depth into the infrared radiated specimen were evidence of this phenomenon. Based on the fact that increasing the source temperature results in shorter overall wavelengths of infrared, a more desirable and efficient system would have employed more filaments with a lower power output but which could be operated at a higher percentage of rated voltage.

The other purpose of this investigation was to determine the physical effects of remelting on UHMWPE. Although at least some surface degradation was noted on all of the infrared treated specimens, physical testing indicated that the bulk of UHMWPE bars was not degraded by either infrared or conventional oven remelting. On the contrary, remelting by both techniques was found to increase some of the key physical properties. Increases in average percent elongation and average impact strength and decreases in swell ratio and percent extractables indicated that remelting enhanced chain entanglement and/or chain interpenetration. Theoretically, achieving higher degrees of chain entanglement and/or chain interpenetration will result in better consolidation. The observed decreases in modulus of remelted UHMWPE bars were probably due to a lower crystallinity, a property that was not measured in this investigation.

The significant decreases in swell ratio and percent extractables after remelting UHMWPE may also have resulted from branching or light cross-linking of polymer chains. In the absence of chemical additives, bond cleavage is necessary to create branching or cross-linking. Based on theory and on the observed degradation at the surface of specimens that were exposed to high doses of infrared radiation in an inert or vacuum environment, it is likely that radiation in the infrared oven and, to a lesser extent, the conventional oven caused bond scission. Calculations show that the energy supplied by infrared radiation is just enough to cause cleavage of C-C and C-H bonds. After these chemical bonds break, the radicals that are formed can be either combined with other radicals, disproportionate with other radicals, combined with a different molecule in side reactions (eg., oxidation), and/or exist indefinitely as free radicals. Both combination and disproportionate are termination reactions that can result in branching or cross-linking. The lower average values of swell ratio and percent extractables for the infrared treated specimens in comparison to the oven remelted specimens, though insignificant, may have been related to the relative intensity of the infrared radiation from the two sources.

Conclusions

Based on the results of this investigation, the following conclusions and recommendations were made:

1. Remelting UHMWPE bar stock using a convention oven or infrared radiation does not appear to degrade the bulk of the material.
2. Remelting UHMWPE by infrared oven and conventional oven increases the average percent elongation, impact strength, while decreasing the modulus in comparison to untreated controls.
3. Remelting UHMWPE by infrared oven and conventional oven decreased the percent extractables and swell ratio in comparison to untreated controls, indicating that the material was better consolidated or that branching or light cross-linking had occurred.

Recommendation

Based on the significant amount of attention that the use of cross-linked UHMWPE in orthopaedic implants has recently received, it is recommended that further studies be conducted to explore the use of infrared radiation to cross-link UHMWPE bar stock.

Inventions

There were no inventions as a result of this CRADA.

Commercialization Possibilities

There were no commercialization possibilities that resulted from this CRADA.

Plans for Future Collaboration

At this time, there are no plans for any future collaboration.

Conclusions

Remelting UHMWPE bar stock using a convention oven or infrared radiation does not appear to degrade the bulk of the material.

Remelting UHMWPE by infrared oven and conventional oven increases the average percent elongation, impact strength, while decreasing the modulus in comparison to untreated controls.

Remelting UHMWPE by infrared oven and conventional oven decreased the percent extractables and swell ratio in comparison to untreated controls, indicating that the material was better consolidated or that branching or light cross-linking had occurred.

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