

ML TDR 64-77

FOREWORD

This report was prepared by the Fibrous Materials Branch and was initiated under Project No. 7320, "Fibrous Materials for Decelerators and Structures," Task No. 732001, "Organic and Inorganic Fibers." The work was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, Miss Joyce C. McGrath, primary project engineer, and Mr. Denver Hale, project engineer in the Materials Physics Division.

The author acknowledges the effort and cooperation of Mr. B. Melvin of the E. I. duPont de Nemours and Company in providing the experimental samples of color sealed yarns for evaluation.

This report covers work from March 1963 to November 1963. Data are recorded in Project Record Book No. 32206.

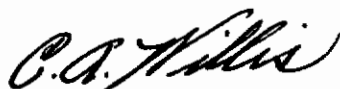
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ABSTRACT

This program involved a comparative study of natural and dyed 100-denier yarns of Nomex (a high temperature resistant fiber formerly known as HT-1) to determine if the inclusion of color in the solution before fiber formation changes or affects the strength or temperature resistant properties of the yarns after exposure to temperatures up to 600° F and/or gamma radiation. A variation in data was obtained between the dyed yarns and the natural color yarns; however this is believed due to a variation in the yarns and not the color process. The color sealed yarns were furnished in very small experimental quantities with zero twist while the natural yarns were furnished from a contract item and had producer's twist. At the higher temperatures, all the yarns had a tendency to become somewhat brittle and fray out. Elongation properties were more readily affected by high temperature and/or gamma radiation than the strength properties.

All data obtained for the color sealed yarns, after exposure to high temperature and/or gamma radiation, indicates that the color sealing process has not adversely affected the strength and high temperature resistant characteristics of the Nomex yarns.

This technical documentary report has been reviewed and is approved.



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INTRODUCTION

The principal advantage of Nomex* (formerly called HT-1), an aromatic, nonmelting polyamide fiber developed by E. I. duPont de Nemours and Company, Inc., over all other known synthetic and natural fibers is its degree of retention and/or improvement of strength properties after exposure to elevated temperature and/or gamma radiation. In appearance the fiber is silvery white with high luster. It maintains a high degree of strength, modulus, and fatigue resistance at temperatures where other natural or synthetic fibers melt or degrade quickly. The fibers do not melt and will not support a flame once the source of the flame is removed, but instead they start to char at temperatures of about 750° F. These fire resistant and nonmelting characteristics will be useful in many applications, such as in personnel decelerators and flight clothing. When these fibers are exposed to temperatures of 450° F, the silvery white color changes to a light yellow and a dark brown color is produced at temperatures in excess of 550° F. The color also gradually darkens with extended periods of exposure to ultraviolet light. These color changes are due primarily to deterioration of the finish (References 1 and 2).

Many applications where Nomex might be utilized, such as in personnel decelerators, require that the material be dyed a certain color or shade for identification or camouflage purposes; however at present Nomex cannot be dyed acceptably by any currently known method. Inasmuch as there is such a requirement, it becomes necessary that a process for dyeing be found. To date all laboratory data has been obtained using Nomex yarns or materials of the natural, silvery white color (References 3 and 4). This data, along with preliminary field test data from various type decelerators, indicates a major improvement over present materials, especially where there is a potential danger from high temperature and/or fire. In order to determine if the properties of dyed Nomex yarns were as good as those of the natural yarns, a comparative evaluation was made using color sealed yarns furnished by E. I. duPont de Nemours and Company, Inc. The yarns were (color sealed) International Orange and Olive Green, two colors used in decelerators. It should be remembered that the color sealed yarns were experimental yarns with zero twist and furnished in very small quantities. The natural yarn was an Air Force Materials Laboratory contract item and had producer's twist. All of the yarns used in the program were 100-denier yarns.

* Registered Trademark of E. I. duPont Company.

EVALUATION PROGRAM

The yarns, one of each of the three colors, were evaluated for strength retention properties only, due to the experimental amount of the color sealed yarn available. Strength retention data was obtained after exposure to thermal and/or gamma radiation for specified periods of time and conditions.

The yarns were submitted to (a) elevated temperatures of 400° F, 500° F, and 600° F for specified periods of time, (b) total gamma radiation dosages of 7.0×10^8 and 1.4×10^9 ergs per gram carbon, (c) total gamma radiation dosages of 7.0×10^8 and 1.4×10^9 in combination with temperatures of 400° F, 500° F, and 600° F, and (d) each of the combinations in (c) above plus two additional hours oven aging at temperatures of 400° F, 500° F, and 600° F.

The two hour elevated temperature exposures were made in circulating hot air oven with automatically controlled temperature and timer devices.

The source of gamma radiation was the 17000 Curie Cobalt-60 source located in the Physics Division of the Air Force Materials Laboratory. Samples of each of the three yarns were irradiated to total dosages of 7.0×10^8 and 1.4×10^9 ergs per gram carbon at normal operating temperature (approx. 100° F) and to the same dosages simultaneously with temperatures of 400° F, 500° F, and 600° F. (Length of time to reach the above dosages was not recorded as total dosage was the prime concern: however the total hours were approximately 20 to 40).

Samples of each of three yarns were exposed to temperatures of 400° F, 500° F, and 600° F for a period of two hours after having been exposed to gamma radiation and also were exposed to gamma radiation simultaneously with temperatures of 400° F, 500° F, and 600° F.

Test specimens for each exposure were wrapped around a card (approx. 2-1/2 inches by 4 inches) made of folded aluminum foil and then placed in an envelope made of aluminum foil, to prevent the specimen from coming in contact with the radiation and temperature sources. All breaking strengths were obtained using an Instron Tensile Tester which records the breaking strength and elongation simultaneously. Both the recorder speed and the rate of crosshead separation on the Instron Tensile Tester was five inches per minute.

CONCLUSION

The dyeing of Nomex yarns has not affected the overall general characteristics of the basic natural color yarns, and data obtained for the 100-denier yarns follows the trend as reported for the 200-denier yarns when tested under similar conditions. Both the color sealed and the natural color yarns showed a tendency to become brittle and "fray out," after exposures to the higher temperatures (500° F and 600° F) and to gamma radiation and temperature, making them difficult to handle. The brittleness and "fraying out" were more apparent in the color sealed yarns than in the natural yarns, probably due to the lack of twist in the color sealed yarns. Variation in data between the natural and the color sealed yarns is believed to be due to the difference in twist and not to factors of the process to include color

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Strength retention of the yarns, both natural and color sealed, under the 500° F exposure conditions was in some instances higher than for the 400° F exposures especially when the yarns were exposed to additional temperatures of 500° F and 600° F and gamma radiation. Elongation results for the natural and Olive Green yarns were more affected by exposure conditions involving temperatures of 400° F and 600° F than by exposure to the 500° F temperature. The elongation data for International Orange yarns, with few exceptions, showed fairly good results under all exposure conditions.

TABLE I
STRENGTH PROPERTIES OF NOMEX YARNS EXPOSED TO ELEVATED TEMPERATURE

	TENACITY (grams/denier)			STRENGTH RETAINED (%)		
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN
ORIGINAL	5.92	5.85	5.58			
ORIGINAL + 2 HRS AT 400° F	5.54	6.03	5.83	93.6	103.07	104.4
ORIGINAL + 2 HRS AT 500° F	5.09	5.50	4.20	85.9	94.0	75.5
ORIGINAL + 2 HRS AT 600° F	2.18	3.29	2.86	37.0	56.3	51.3

TABLE 2
ELONGATION PROPERTIES OF NOMEX YARNS EXPOSED TO ELEVATED TEMPERATURE

	ELONGATION (%)			ELONGATION RETAINED (%)		
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN
ORIGINAL	14.2	11.2	12.0			
ORIGINAL + 2 HRS AT 400° F	12.3	12.6	12.0	86.6	112.5	100.0
ORIGINAL + 2 HRS AT 500° F	10.3	11.6	7.6	72.5	103.5	63.3
ORIGINAL + 2 HRS AT 600° F	2.2	5.5	3.9	15.5	49.2	32.5

TABLE 3
STRENGTH PROPERTIES OF NOMEX YARNS EXPOSED TO GAMMA RADIATION AND/OR
ELEVATED TEMPERATURE (400°F)

	TENACITY (grams/denier)			STRENGTH RETAINED (%)		
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN
ORIGINAL	5.92	5.85	5.58			
ORIGINAL + 2 HRS AT 400°F	5.54	6.03	5.83	93.6	103.07	104.4
GAMMA, 7.0 x 10 ⁸ *	5.32	5.95	5.69	89.8	101.7	101.9
GAMMA, 7.0 x 10 ⁸ + 2 HRS AT 400°F	6.22	6.46	5.70	105.06	110.4	102.1
GAMMA, 7.0 x 10 ⁸ /400°F	5.30	5.54	5.81	89.5	94.7	104.1
GAMMA, 7.0 x 10 ⁸ /400°F + 2 HRS AT 400°F	5.40	5.82	5.51	91.4	99.5	98.8
GAMMA, 7.0 x 10 ⁸ /400°F + 2 HRS AT 500°F	4.41	5.26	2.38	74.5	89.9	42.7
GAMMA, 7.0 x 10 ⁸ /400°F + 2 HRS AT 600°F	2.56	2.87	2.38*	43.3	49.1	42.7
GAMMA, 1.4 x 10 ⁹ *	5.71	5.75	5.62	96.5	98.3	100.7
GAMMA, 1.4 x 10 ⁹ + 2 HRS AT 400°F	5.58	5.98	5.58	94.3	102.2	100.0
GAMMA, 1.4 x 10 ⁹ /400°F	4.76	5.54	3.31	80.5	94.7	59.3
GAMMA, 1.4 x 10 ⁹ /400°F + 2 HRS AT 400°F	4.02	5.04	2.98	67.9	86.2	53.4
GAMMA, 1.4 x 10 ⁹ /400°F + 2 HRS AT 500°F	5.11	4.25	2.38	86.4	72.7	42.7
GAMMA, 1.4 x 10 ⁹ /400°F + 2 HRS AT 600°F	3.47	3.73	2.92**	58.6	63.8	52.5

* NORMAL OPERATING TEMPERATURE, APPROX. 100°F

** FRAYED AND BRITTLE

NOTE: /°F INDICATES GAMMA AND TEMPERATURE SIMULTANEOUSLY

+ HOURS AT °F INDICATES OVEN AGING

TABLE 4
STRENGTH PROPERTIES OF NOMEX YARNS EXPOSED TO GAMMA RADIATION AND/OR
ELEVATED TEMPERATURE (500°F)

	TENACITY (grams/denier)			STRENGTH RETAINED (%)		
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN
ORIGINAL	5.92	5.85	5.58			
ORIGINAL + 2 HRS AT 500°F	5.09	5.50	4.20	85.9	94.0	75.5
GAMMA, 7.0 x 10 ⁸	5.32	5.95	5.69	89.9	101.7	101.9
GAMMA, 7.0 x 10 ⁸ + 2 HRS AT 500°F	4.60	4.74	2.0	77.7	81.9	35.9
GAMMA, 7.0 x 10 ⁸ /500°F	4.68	5.0	3.14	79.1	85.5	56.3
GAMMA, 7.0 x 10 ⁸ /500°F + 2 HRS AT 400°F	4.52	4.9	2.76	76.4	83.8	49.5
GAMMA, 7.0 x 10 ⁸ /500°F + 2 HRS AT 500°F	4.34	4.8	3.36**	73.4	82.1	60.3
GAMMA, 7.0 x 10 ⁸ /500°F + 2 HRS AT 600°F	3.43	3.69	3.13*	57.9	63.1	56.1
GAMMA, 1.4 x 10 ⁹	5.71	5.75	5.62	96.5	98.3	100.7
GAMMA, 1.4 x 10 ⁹ + 2 HRS AT 500°F	4.43	4.83	2.69	74.8	82.6	48.2
GAMMA, 1.4 x 10 ⁹ /500°F	3.67	4.89	4.52	62.0	83.6	81.1
GAMMA, 1.4 x 10 ⁹ /500°F + 2 HRS AT 400°F	4.24	4.72	4.06	71.6	80.7	72.8
GAMMA, 1.4 x 10 ⁹ /500°F + 2 HRS AT 500°F	4.19	4.46	4.20**	70.8	76.1	75.3
GAMMA, 1.4 x 10 ⁹ /500°F + 2 HRS AT 600°F	3.58*	3.93*	3.55*	60.5	67.9	63.7

* FRAYED AND BRITTLE

** FRAYED

TABLE 5
STRENGTH PROPERTIES OF NOMEX YARNS EXPOSED TO GAMMA RADIATION/AND OR
ELEVATED TEMPERATURE (600°F)

	TENACITY (grams/denier)			STRENGTH RETAINED (%)		
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN
ORIGINAL	5.92	5.85	5.58			
ORIGINAL + 2 HRS AT 600°F	2.18	3.29	2.86	37.0	56.3	51.3
GAMMA, 7.0 x 10 ⁸	5.32	5.95	5.69	89.9	101.7	101.9
GAMMA, 7.0 x 10 ⁸ + 2 HRS AT 600°F	2.93	3.06	2.40	49.5	52.3	43.1
GAMMA, 7.0 x 10 ⁸ /600°F	2.77	3.54	2.31	46.8	60.6	41.6
GAMMA, 7.0 x 10 ⁸ /600°F + 2 HRS AT 400°F	2.97	3.71	3.10	50.2	63.5	55.6
GAMMA, 7.0 x 10 ⁸ /600°F + 2 HRS AT 500°F	2.75	3.12	2.50**	46.8	53.4	44.8
GAMMA, 7.0 x 10 ⁸ /600°F + 2 HRS AT 600°F	2.09	3.04	2.65*	35.7	51.9	47.6
GAMMA, 1.4 x 10 ⁹	5.71	5.75	5.62	96.5	98.3	100.7
GAMMA, 1.4 x 10 ⁹ + 2 HRS AT 600°F	2.74	2.74	2.54	46.2	46.8	45.5
GAMMA, 1.4 x 10 ⁹ /600°F	2.13	1.05*	1.04*	36.0	17.9	18.7
GAMMA, 1.4 x 10 ⁹ /600°F + 2 HRS AT 400°F	2.07	2.22	1.77**	34.8	37.5	31.8
GAMMA, 1.4 x 10 ⁹ /600°F + 2 HRS AT 500°F	2.30	2.35	2.12*	38.9	40.2	38.0
GAMMA, 1.4 x 10 ⁹ /600°F + 2 HRS AT 600°F	1.89*	2.41*	2.15*	31.9	41.2	36.6

* FRAYED AND BRITTLE

** FRAYED

TABLE 6
 ELONGATION PROPERTIES OF NOMEX YARNS EXPOSED TO GAMMA RADIATION AND/OR
 ELEVATED TEMPERATURE (400°F)

	ELONGATION (%)				ELONGATION RETAINED (%)			
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN	ELONGATION RETAINED (%)	
							NATURAL	INT. ORANGE
ORIGINAL	14.2	11.2	12.0					
ORIGINAL + 2 HRS AT 400°F	12.3	12.6	12.0	86.6	112.5	100.0		
GAMMA, 7.0 x 10 ⁸	11.5	14.1	13.2	81.0	125.9	110.0		
GAMMA, 7.0 x 10 ⁸ + 2 HRS AT 400°F	16.2	14.9	11.5	114.1	133.0	95.8		
GAMMA, 7.0 x 10 ⁸ / 400°F	11.2	12.1	12.9	78.9	108.1	107.5		
GAMMA, 7.0 x 10 ⁸ / 400°F + 2 HRS AT 400°F	11.8	12.2	11.6	83.1	108.9	96.5		
GAMMA, 7.0 x 10 ⁸ / 400°F + 2 HRS AT 500°F	7.8	10.5	2.5	54.9	93.7	20.9		
GAMMA, 7.0 x 10 ⁸ / 400°F + 2 HRS AT 600°F	3.3	4.2	2.8	23.3	37.5	23.3		
GAMMA, 1.4 x 10 ⁹	15.0	12.9	13.7	105.6	115.2	114.1		
GAMMA, 1.4 x 10 ⁹ + 2 HRS AT 400°F	13.5	13.9	13.7	95.1	124.1	114.1		
GAMMA, 1.4 x 10 ⁹ / 400°F	8.5	10.6	4.3	59.9	94.6	35.8		
GAMMA, 1.4 x 10 ⁹ / 400°F + 2 HRS AT 400°F	6.3	9.8	3.4	44.4	87.5	28.4		
GAMMA, 1.4 x 10 ⁹ / 400°F + 2 HRS AT 500°F	3.5	7.3	2.8	24.6	65.2	23.3		
GAMMA, 1.4 x 10 ⁹ / 400°F + 2 HRS AT 600°F	6.5	7.1	4.6	45.8	63.4	38.4		

TABLE 7
 ELONGATION PROPERTIES OF NOMEX YARNS EXPOSED TO GAMMA RADIATION AND/OR
 ELEVATED TEMPERATURE (500°F)

	ELONGATION (%)			ELONGATION RETAINED (%)		
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN
ORIGINAL	14.2	11.2	12.0			
ORIGINAL + 2 HRS AT 500°F	10.3	11.6	7.6	72.5	103.5	63.3
GAMMA, 7.0 x 10 ⁶	11.5	14.1	13.2	81.0	125.9	110.0
GAMMA, 7.0 x 10 ⁸ + 2 HRS AT 500°F	8.3	8.8	2.4	58.5	78.6	20.0
GAMMA, 7.0 x 10 ⁹ / 500°F	10.3	10.3	4.2	72.5	93.7	35.0
GAMMA, 7.0 x 10 ⁸ / 500°F + 2 HRS AT 400°F	8.4	8.6	3.6	59.2	76.8	30.0
GAMMA, 7.0 x 10 ⁸ / 500°F + 2 HRS AT 500°F	8.7	10.5	5.3	61.3	95.1	44.2
GAMMA, 7.0 x 10 ⁸ / 500°F + 2 HRS AT 600°F	6.6	7.8	5.3	46.5	69.7	44.2
GAMMA, 1.4 x 10 ⁹	15.0	12.9	13.7	105.6	115.2	114.1
GAMMA, 1.4 x 10 ⁹ + 2 HRS AT 500°F	8.8	10.5	3.7	61.8	95.1	30.8
GAMMA, 1.4 x 10 ⁹ / 500°F	6.4	10.8	9.6	45.1	96.4	80.8
GAMMA, 1.4 x 10 ⁹ / 500°F + 2 HRS AT 400°F	8.6	11.4	7.7	60.6	101.8	62.2
GAMMA, 1.4 x 10 ⁹ / 500°F + 2 HRS AT 500°F	8.8	9.9	9.6	61.8	87.5	80.8
GAMMA, 1.4 x 10 ⁹ / 500°F + 2 HRS AT 600°F	7.3	11.0	9.0	44.4	98.2	75.0

TABLE 8
 ELONGATION PROPERTIES OF NOMEX YARNS EXPOSED TO GAMMA RADIATION AND/OR
 ELEVATED TEMPERATURE (600°F)

	ELONGATION (%)				ELONGATION RETAINED (%)				
	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN	NATURAL	INT. ORANGE	OLIVE GREEN
ORIGINAL	14.2	11.2	12.0						
ORIGINAL + 2 HRS AT 600°F	2.2	5.5	3.9	15.5	49.1	32.5			
GAMMA, 7.0 x 10 ⁶	11.5	14.1	13.2	81.0	125.9	110.0			
GAMMA, 7.0 x 10 ⁸ + 2 HRS AT 600°F	5.1	5.7	3.8	35.9	50.9	31.7			
GAMMA, 7.0 x 10 ⁸ /600°F	4.5	6.8	3.4	31.8	60.7	28.3			
GAMMA, 7.0 x 10 ⁸ /600°F + 2 HRS AT 400°F	5.3	7.6	5.0	37.3	67.8	41.7			
GAMMA, 7.0 x 10 ⁸ /600°F + 2 HRS AT 500°F	4.8	5.2	3.4	33.8	46.4	28.3			
GAMMA, 7.0 x 10 ⁸ /600°F + 2 HRS AT 600°F	3.1	5.9	5.2	21.8	52.7	43.3			
GAMMA, 1.4 x 10 ⁹	13.5	12.9	13.7	95.1	115.2	114.1			
GAMMA, 1.4 x 10 ⁹ + 2 HRS AT 600°F	4.3	4.2	3.8	30.3	37.5	31.7			
GAMMA, 1.4 x 10 ⁹ /600°F	3.2	6.0	1.7	22.5	53.6	14.2			
GAMMA, 1.4 x 10 ⁹ /600°F + 2 HRS AT 400°F	3.9	3.9	2.6	27.5	34.2	21.7			
GAMMA, 1.4 x 10 ⁹ /600°F + 2 HRS AT 500°F	4.0	4.6	3.6	28.2	41.1	30.0			
GAMMA, 1.4 x 10 ⁹ /600°F + 2 HRS AT 600°F	3.6	5.1	3.6	26.8	45.5	30.0			

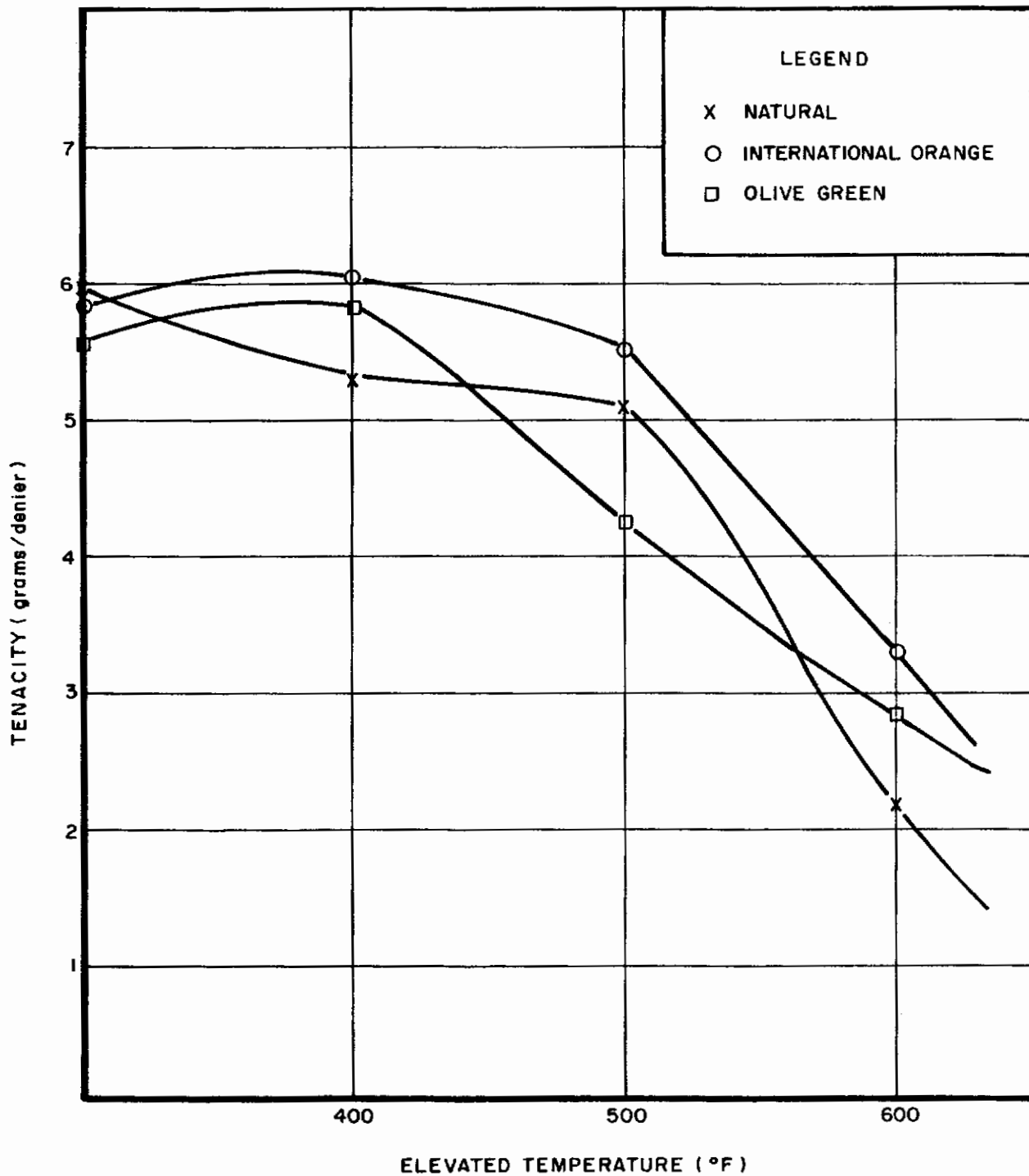


Figure 1. Tenacity of Nomex Yarns After 2 Hours at Elevated Temperature

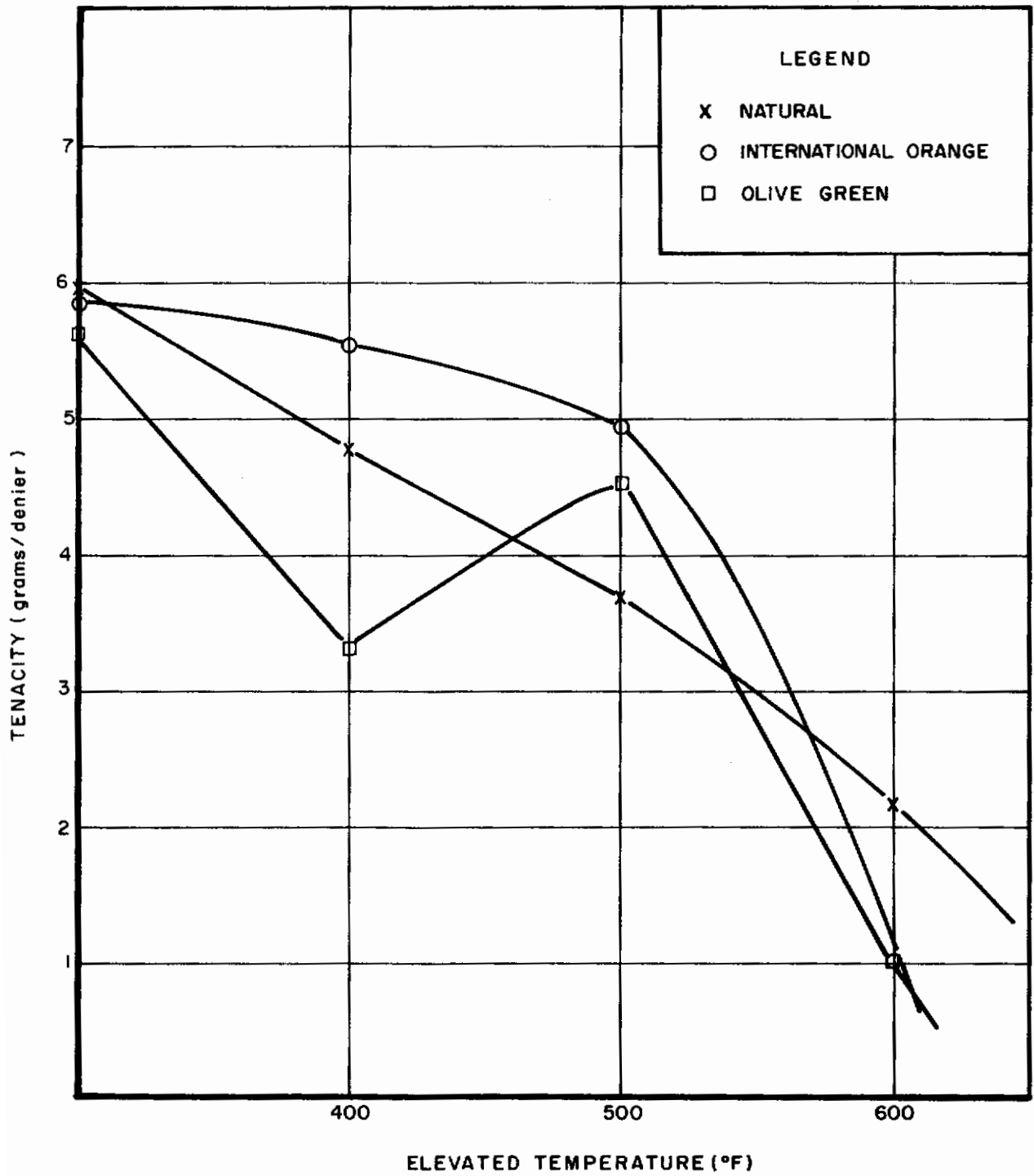


Figure 2. Tenacity of Nomex Yarns Exposed to Gamma Radiation (1.4×10^9) at Elevated Temperature

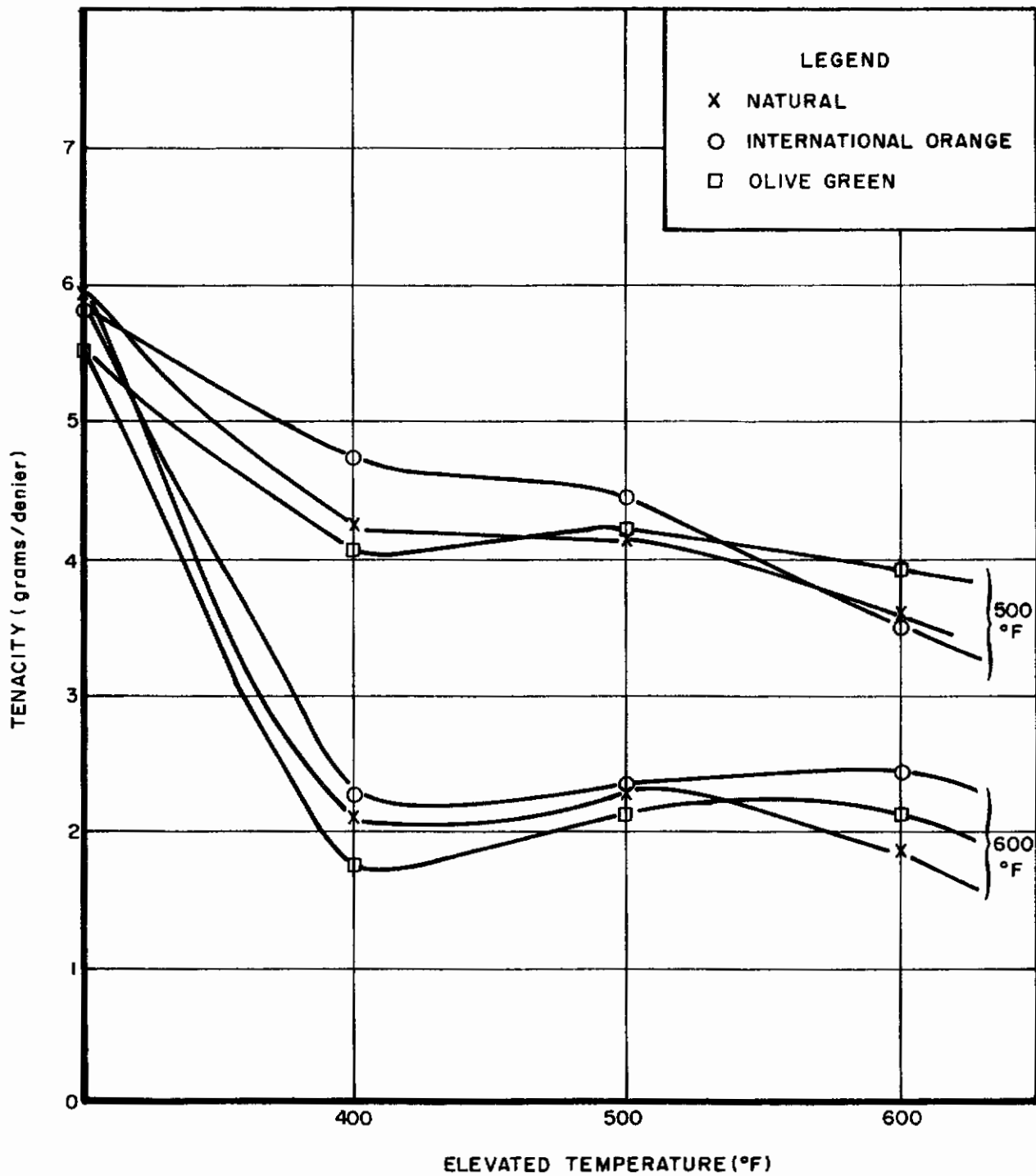


Figure 3. Tenacity of Nomex Yarns Exposed to Gamma Radiation (1.4×10^9) at 500°F and 600°F Plus 2 Hours Elevated Temperature

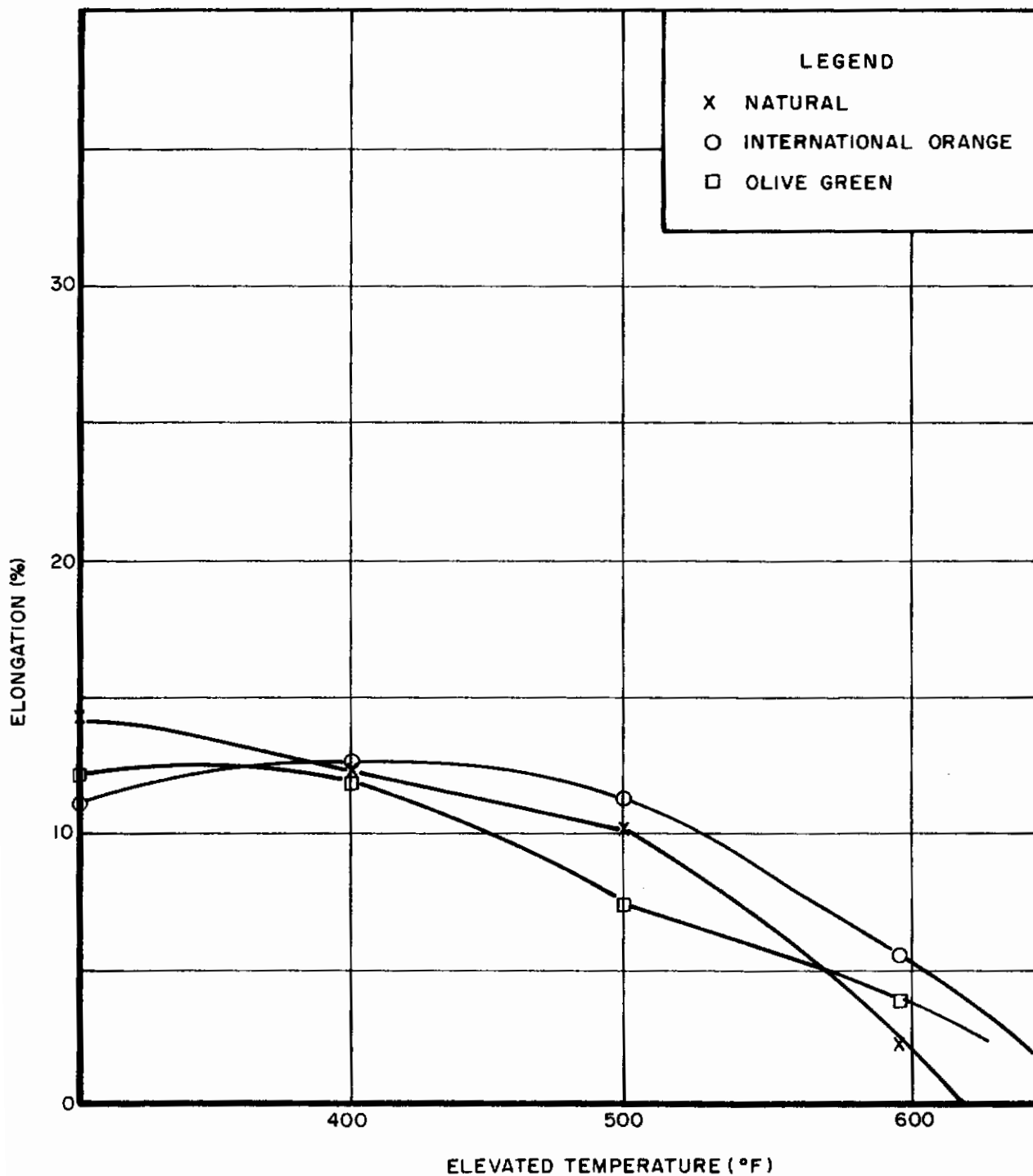


Figure 4. Elongation of Nomex Yarns After 2 Hours at Elevated Temperature

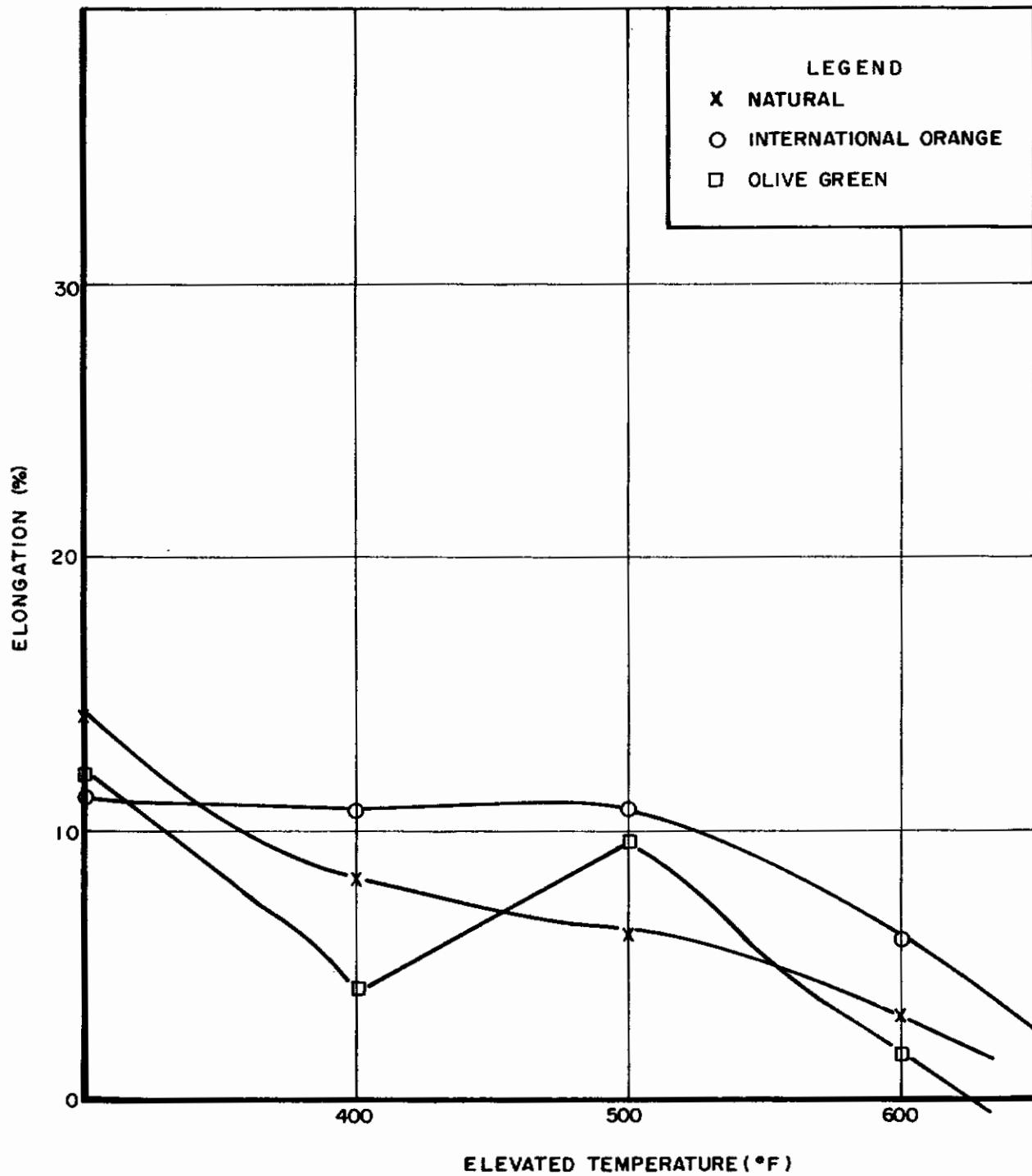


Figure 5. Elongation of Nomex Yarns Exposed to Gamma Radiation (1.4×10^9) at Elevated Temperature

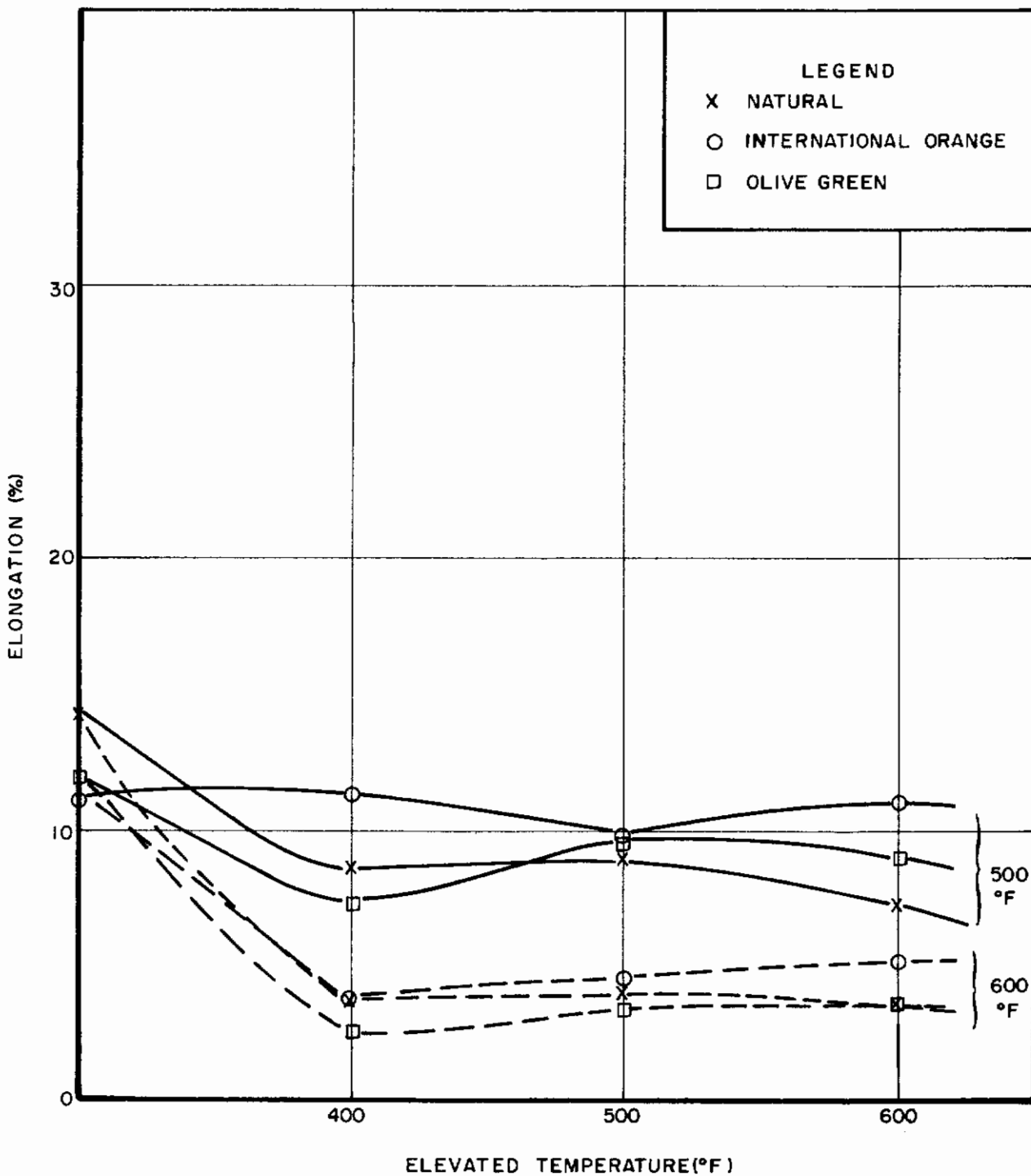


Figure 6. Elongation of Nomex Yarns Exposed to Gamma Radiation (1.4×10^9) at 500°F and 600°F Plus 2 Hours at Elevated Temperature

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