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Detective Work with High Speed Testing: The Location of a Phase Change Area

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Synopsis

High speed tensile tests on Lexan polycarbonate resin were carried out on three grades of material at four test speeds from 200 to 15,000 in./min. and at five temperatures from −30 to +140°C. A consideration of the results of these tests led to a number of interesting conclusions. Among these conclusions was the identification of a plateau in the plots of some of the variables vs temperature in the 125° to 140°C. range. A more complete study of the variation in the tensile yield values of one of the grades of Lexan resin at 2 and 20 in./min. testing speed over the temperature range, and somewhat beyond, has been made to define this unusual effect.

The fact that Lexan polycarbonate resin is ductile at room temperature at testing speeds up to 12,000 in./min. was clearly brought out by some tests that Mel Silberberg kindly ran for us on a Plastechon machine at an ASTM meeting in Atlantic City in June 1960. This has again been corroborated recently in the second report of the ASTM Committee D-20 Task Group on High Speed Testing authored by Gordon D. Patterson, Jr.

However, recently we decided to make a study of the tensile properties of three grades of Lexan resin over a range of temperature from −30 to +140°C. and of testing speed from 200 to 15,000 in./min. This work was carried out by the Plas-Tech Equipment Company on their Plastechon 591 hydraulic universal tester.

The conditions for these tests were as follows: (*1*) we assumed that despite the fact that the three grades of Lexan resin were different in one way or another that the tensile test is a poor measure of minor changes in molecular weight and, therefore, one sample of each grade could be run at each set of conditions as though the three grade samples were three samples of the same grade; (*2*) the samples used were ASTM Type I injection molded tensile bars, all molded under essentially the same conditions; (*3*) the temperatures to be used were −30, +23, +80, +125, and +140°C.; (*4*) samples to be tested at 80 and at 125°C. were to be conditioned at 80 and at 125°C., respectively, in an air circulating oven overnight before testing. The samples to be tested at 140°C. were to be conditioned at 140°C. for 5 hr. in an air circulating oven before testing; (*5*) an average preconditioning time of about 45 min. at the test temperature ±1° C. was used before each load application; (*6*) the rates of loading used were: 200, 2000, 6000 and 15,000 in./min. A velocity trace was made with a test sample mounted in the grips at each speed to determine the rise time and the amount of slack to be allowed for. The tests were then conducted on the Plastechon 591 machine using a mechanical slack adapter which allowed the piston to attain the desired operating velocity before contacting the specimen; and (7) one test on each grade was then run at the set rate of loading starting with the lowest temperature and moving up to 140°C. in the prescribed steps. The velocity was then changed to the next higher rate of loading. The rise time and slack allowance was determined for this new speed and the tests run off from the lowest up to the highest temperature, and so on.

Now, what type of information was collected? Five bits of data were obtained for each sample: The tensile strength at yield and at failure, the elongation in inches at yield and at failure, and the work to break or the area under the stress-strain curve. Thus, for one sample each of three grades of resin, five temperatures, four rates of loading and five pieces of data per sample, we had a total of 300 entries to organize and analyze.

We used two methods of analysis. The first, in which the variances of the five items of data were calculated and judged for their significance by the F test, was suggested by our Dr. J. J. Keane. The second, in which data from the first analysis was used to analyze the practical effect of the variable, was proposed by G. J. Hahn of our General Engineering Laboratory.

The analysis of the variances by the F test indicated the following:

TABLE I
Analysis of the Variances by the F Test

Variable or interaction	Significant at % level				
	Tensile Yield		Tensile Ultimate		Work to Break
	Strength	Elongation	Strength	Elongation	
Temperature (A)	1	1	1	1	N.S.
Rate of loading (B)	5	5	N.S.	1	N.S.
Grade (C)	N.S.[a]	N.S.	1	1	5
AB interaction	1	N.S.	N.S.	N.S.	N.S.
AC interaction	N.S.	N.S.	N.S.	5	N.S.
BC interaction	N.S.	N.S.	N.S.	N.S.	N.S.

[a] N.S. means not significant statistically.

From Table I, we can see that temperature is a very significant factor in all results except work to break. The rate of loading is significant at the 1% level only for tensile elongation at failure. This factor affects the tensile yield values only at a 5% level of significance. Our assumptions with regard to the low worth of tensile data in differentiating between grades is not at all borne out in the effect of the variation in grade when the effect of variation in grade on the ultimate tensile strength and elongation and the work to break is considered. We will see the reason for this clearly in the second analysis of data.

The variable interaction effects were significant in only two cases as shown in the table. Otherwise, they were of no statistical significance.

From the above analysis of variance, we are able to see what factors are significant and at what level, but we are not able to see what trend in the data the variables that are significant really generate. For such information, we must go to the second analysis. Here, the data for each of the five results obtained per sample point, were treated in the following ways: *(1)* the data for each grade were averaged over the four rates of loading at each temperature; *(2)* the data for each grade were averaged over the five temperatures for each rate of loading; *(3)* the data for each rate of loading were averaged over the three grades for each temperature; and *(4)* the range of values over the three grades was presented for the four rates of loading and the five temperatures.

The above considerations led to twenty tables of which some are of more interest than others. With these tables, we can begin to put some trend values and practical meaning into the significance levels found in the analysis of variances. We will also be able to begin some detective work based on a finding in these tables which the first analysis does not bring out at all. We will discuss both of these points next.

Previously, we had mentioned that our assumption that all three grades were the same as far as tensile properties were concerned was found to be in error based on the data for the ultimate tensile strength and elongation. This can be clearly seen in Tables II and III.

Here we see that Lexan 130-111 material is definitely lower in ultimate tensile strength than the other two. Further, in the lower compilation, we see that the ultimate elongation of this same material is less than for the other two. Thus, while the analysis of variance indicates that the grade is a significant factor, we can now amplify this generalization by saying that

TABLE II

Ultimate Tensile Strength, psi $\times 10^{-3}$, Averaged Over the Four Rates of Loading for the Grades and Temperatures

Temp., °C.	130-111	101-112	101-111	Average
−30	12.15	11.80	11.51	11.82
23	9.83	11.10	10.98	10.64
80	7.99	9.33	9.83	9.05
125	6.57	7.65	7.52	7.25
140	6.57	7.38	8.46	7.47
Average	8.62	9.45	9.66	9.24

TABLE III
Elongation at Failure, Inches, Averaged Over the Five Temperatures for the Grades and Rates of Loading

Rate of loading, in./min.	130-111	101-112	101-111	Average
200	3.00	4.72	4.47	4.06
2,000	2.97	4.76	4.30	4.01
6,000	2.50	3.46	3.59[a]	3.18[a]
15,000	3.56	3.82	3.68	3.69
Average	3.01	4.19	4.07	3.74

[a] One sample broke brittlely (6000 in./min. at 125°C.)

the Lexan 130-111 grade, which is the high melt viscosity material, has a significantly lower ultimate tensile strength and elongation than the Lexan 101 grade materials which have lower melt viscosity.

In a similar manner we can study the trend in the effect of each of the variables on the results obtained and come up with the following conclusions:

(*1*) The effect of temperature is very marked. An increase in the temperature brings a reduction in the tensile strength at yield and at failure. The elongation at yield decreases with increasing temperature, while the elongation at failure tends to increase with increasing temperature. The temperature has little effect, on the other hand, on the work to break.

(*2*) The rate of loading has a much lower effect than temperature on the results. There seems to be a peak in the tensile yield and ultimate strengths somewhere between 2000 and 6000 in./min. A minimum in the elongation at yield occurs in the same rate of loading region. The elongation at failure which is a significant factor at the 1% level decreases as the rate of loading increases. The work to break is not influenced by the changes in the rate of loading.

The effect of the different grades has been discussed above. But, we can summarize this effect here by saying that the effect of the grade on the tensile yield and elongation, as well as on the work to break, is negligible. In the tensile strength at failure, the high melt viscosity material is lowest, as is the value of the elongation at failure. This leads to a lower work to break value for this material as well.

Now, having gotten a lot of background out of the way, we can proceed to the other aspect of this examination. In work on Lexan molded parts in the past, we had found that if these parts are heated at 125°C. in an air circulating oven for at least 3–4 hr., some profound changes in physical properties take place. For example, the modulus of elasticity increases; the surface hardness goes up about 4 points on the Rockwell M scale; the critical thickness in notched impact strength decreases, etc. All of the above changes in properties were noted by tests conducted at room temperature after the heating at elevated temperatures.

During our analysis of data, such as in Table IV, we realized that the effect of this phenomenon was in evidence even in the tests run at elevated temperatures. As you can see in Table III, the values of the tensile yield strength, averaged over the rates of loading, decrease to a minimum at about 125°C. and then level off or go up slightly at 140°C. This is a very significant finding because this means that while the yield strength drops from about 11000 psi at room temperature to about 8500 psi at 125°C., Lexan parts do not change in strength from 125°C. at least up to 140°C. A remarkable phenomenon!

TABLE IV
Tensile Yield Strength Values, psi $\times$ 10^{-3}, Averaged Over the Four Rates of Loading for Each Grade at Each Temperature

Temp., °C.	130-111	101-112	101-111	Average
−30	13.45	12.40	13.23	13.03
23	10.54	11.15	10.60	10.76
80	8.93	9.25	9.16	9.11
125	8.07	8.38	9.29	8.58
140	8.55	8.59	9.23	8.79
Average	9.91	9.95	10.30	10.05

We then examined the rest of the data tables and found that the elongation at yield also exhibited somewhat the same pattern with temperature. The minimum in this case started about 80°C. and even increased up to 140°C. The ultimate tensile strength shows about the same trend as the yield strength, as does the elongation at failure. The work to break does not seem to be affected at all in this manner by temperature changes. As far as this variable is concerned, the analysis of variance indicated that the error factor was about half the total variance. This might be interpreted to mean that the work to break was very imprecisely determined in this study as compared to the other results obtained.

Having found this phenomenon in the high speed data, we then proceeded to examine it in some detail using lower speeds and our Instron tester. We felt that we could safely go to the much lower rates of loading for this study because it had been shown in the high speed study that the rate of loading was not of high significance as a variable as far as tensile yield strength was concerned.

In order to better define the dimensions of the tensile yield strength plateau, we determined the yield values at both 2 and 20 in./min. crosshead speed at 25, 50, 75, 90, 100, 110, 120, 125, 130, 140, and 150°C. using at least three samples per point. As before, the samples were conditioned overnight at the temperature of test up to and including 130°C. The 140 and 150°C. runs were made on samples preconditioned at these temperatures for four hours. The data obtained are given in Figure 1.

Here, we can clearly see that the plateau begins about 90°C. and extends to about 120°C. for the 2 in./min. rate of loading. At the 20 in./min.

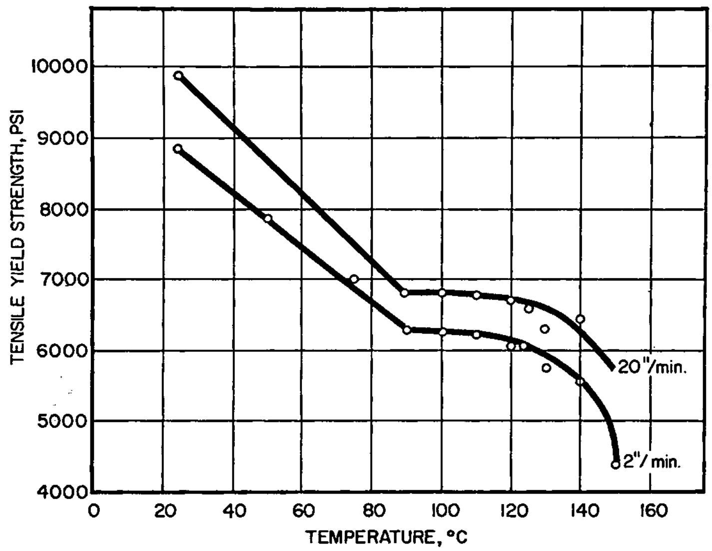

Fig. 1. Dimensions of the tensile yield strength.

rate, the plateau extends out to about 130°C. before beginning to fall off rapidly. The effect of the speed of loading on the length of the plateau which brings about the greater length at the higher speed is already predicted in the previous high speed testing results where, if you will recall, at these speeds the plateau extended out to at least 140°C. even at 200 in./min. rate of loading. The above data were obtained on the Lexan 130-111 grade material of the same lot as used previously.

I wish that I could conclude this paper with a brilliant explanation of the results that I have shown you and leave you with a clear picture of the cause of the phenomenon that we have found in Lexan polycarbonate resin. We have studied many possible leads such as x-ray diagrams taken before and after heating, specific volume measurements up and down the temperature range, nuclear magnetic resonance studies, etc., but we have no clue as yet as to what is taking place.

One valuable bit of information we have found: the phenomenon is reversible. That is, after a sample has been heated at 125°C. for enough hours to, say, increase the Rockwell hardness, the original hardness value can be recovered by heating the sample over T_g, say, to 160°C., for a short time, and cooling it quickly to room temperature. If desired, the whole procedure, then, can be repeated.

Therefore, to summarize what we have discussed today, we have presented some high speed testing data on tensile properties of Lexan polycarbonate resins of three different grades over a temperature range of −30 to 140°C. using speeds of testing from 200 to 15,000 in./min. which lead to discovery of a plateau of tensile strength vs temperature. The extent of this plateau was explored more fully to define its extent. However, no explanation of the phenomenon is available to date. It appears that we may need a real Hawkshaw to decipher this one.

Résumé

Des essais d'élongation à vitesse élevée sur une résine de polycarbonate Lexan ont été effectués pour trois sortes de matériel à quatre vitesses d'essai de 200 à 15000 in/min et à cinq températures de −30 à +140°C. L'examen des résultats de ces tests conduit à un nombre de conclusions intéressantes. Parmi ces conclusions on peut identifier un plateau dans les diagrammes de certaines des variables en fonction de la température dans le domaine de 125° à 140°C. Une étude plus complète de la variation des valeurs de rendements de l'élongation d'une des sortes de résine Lexan à une vitesse d'essai de 2 et 20 in/min dans le domaine de température et quelque peu au delà, a été efféctuée pour définir cet effet inhabituel.

Zusammenfassung

Hochgeschwindigkeit- Zugtests mit Lexan-Polycarbonatharz wurden mit drei Materialsorten und vier Testgeschwindigkeiten von 200 bis 15000″ pro Minute und bei fünf Temperaturen von −30 bis +140°C ausgeführt. Eine Auswertung der Testergebnisse führte zu einer Reine von interessanten Folgerungen. Eine dieser Folgerungen war die Identifizierung eines Plateaus in den Kurven einiger Variabler gegen die Temperatur im Bereich von 125° bis 140°C. Eine vollständigere Untersuchung der Zugfestigkeitswerte einer Sorte von Lexanharz bei 2 und 20″ pro Minute Testgeschwindigkeit über diesen Temperaturbereich und etwas darüber, wurde zur Bestimmung dieses ungewöhnlichen Effekts ausgeführt.