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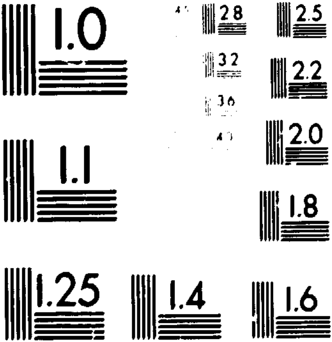

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1985

FINAL REPORT

Dr T.NAGABHUSHANAM
Chief Technical Adviser
UNIDO PROJECT ST/BRA/81/T01

Brazil. DEVELOPMENT OF CARBON FIBER TECHNOLOGY.

(July 1982 - Dec 1984)

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A WORD OF THANKS

I thank Prof. E.Fitzer,Special Technical Adviser, for having confidence in me, for introducing to this Project and for his help in assisting me to solve all the problems related to the Project.

I thank Mr M.A.Youseff, UNIDO Inter-regional Adviser for his timely cooperation, assistance from his side whenever I looked for it.

I thank Mr Niels Brandt, Chief, Section for Latin America and the Caribbean for his cooperation, moral support.

I thank Mr P.Koenz, Resident Representative and his staff for their constant and immediate assistance to the Project and to me.

I thank Dr A.Soto Krebs, SIDFA and his staff for their constant assistance and cooperation.

I thank T.Cel Kessel, National Project Coordinator, for his keen involvement in the Project activities, providing the required facilities and for creating a congenial atmosphere in the group.

I thank Capt Scyllas Souza da Costa, the Carbon Fiber Group Leader, for his personal involvement in the Project and for showing utmost enthusiasm to provide all that required at his level to the bench workers in the group.

Lastly, though not least, I thank all the counterpart scientists involved in the Project, who listened to me, cooperated with me, appreciated the objectives of the Project and did their best in achieving the objectives of the Project.

PERSONAL COMMENTS

During my 30 months stay and especially at the time of beginning when I joined the Project, Prof. Fitzer, the Special Technical Adviser of the Project had been mainly instrumental in advising me in all aspects of the Project. His constant advise and assistance had been immense use to me in discharging my duties. His scientific consultations to us and introductions to the experts and industrialists and industries in the developed countries had a positive effect on the project. What success has been achieved in this Project could not have been achieved without his timely advice and assistance.

Eventhough things were not clear at the beginning of 1982, with the induction of T.Cel Kessel as National Chief of the Project, the situation has completely changed. T.Cel Kessel has personally involved in the Project activities in spite of his busy activities as the Chief of the Division. It is he, who managed to get whatever needed for the Project. Without his timely solving of the Project problems, the Project would not have achieved its goals.

Above all, the credit should go to the counterpart scientific Group Leader Capt Scyllas Souza da Costa and his group. These are the main people who absorbed the shocks from the administrative and technical experts and welcomed the suggestions and were always cooperative. I should say it is an excellent group that CTA/PMR could have. The maximum credit should go to these bench workders. I had excellent cooperations from Capt Scyllas Souza da Costa and all the sub-group chiefs and individuals who were involved in the Project.

FISIBA people who were involved in the precursor developmental activities, especially Vicente Nonato de Sá was very cooperative to me and to our Project.

UNIDO office in Vienna and Brasília gave me extreme cooperation . Mr Youseff, the Senior Inter-regional Adviser gave me excellent support in discharging my duties. Mr Soto Krebs had always been at our doors to help us in our local problems, and his collegue Detlev's assistance was utmost importance.

The UNDP office, Resident Representative, Mr Peter Koenz and his associates were constantly assisting us in all requirements.

1.0- POSITION AT THE TIME OF ARRIVAL

I arrived at the Project site Centro Técnico Aeroespacial (CTA), São José dos Campos, São Paulo, on 1st July 82. My job activities are mainly to train the Project staff in precursor work, polymerization and to act as Chief Technical Adviser of the Project.

Position of the Group

At the time of my arrival, there were:

- No polymerization activities
- No spinning activities
- No lab. facilities to conduct experiments
- No building facilities for precursor group and surface treatment group and Technical personnel were insufficient.

Eventhough, research activities were going on, heat treatment no systematic experiments were planned and conducted. Only one step oxidation and one step carbonization was going on using CTA made heat treatment equipment. Characterization of the raw materials and end products was lacking. The developmental activities after my arrival are herewith brought out year wise.

2.0- DEVELOPMENTAL ACTIVITIES IN THE PRECURSOR WORK

2.1- Activites in 1982

i - Polymerization Studies

For the first time, the group was trained in the polymerization of acrylonitrile in the presence of comonomers like methylacrylate in aqueous medium. Free radical polymerization methods were introduced. Initiator system such as potassium persulfate-bisulfite was used to polymerize the vinylmonomers. The polymers synthesized were purified and characterized by viscometric methods outside PMR/CTA as there were no facilities at that time in CTA.

Table 1 gives some of the results of polymerizations. Meanwhile, in August 1982, the lab. model wet spinning assembly arrived (supplied by UNIDO) at the Project site. As the spinning experiments require larger quantity of the polymers, and as CTA group had only a small polymerization vessals, ie 100 to 200 grams capacity and had no comonomers, it is felt important to standardize the spinning experiments using Fibras Sintéticas da Bahia S.A. (FISIBA) supplied commercial acrylic polymers. The polymerization experiments were postponed to a later date. The activities were concentrated on spinning of acrylic fibers, in order to standardize the spinning aparameters.

Table 1 - Results of polymerization

POLYMER Nº	REDUCED VISCOSITY
1	4.46
2	2.45
3	2.21
4	2.89
5	2.84
6	3.19
7	6.06

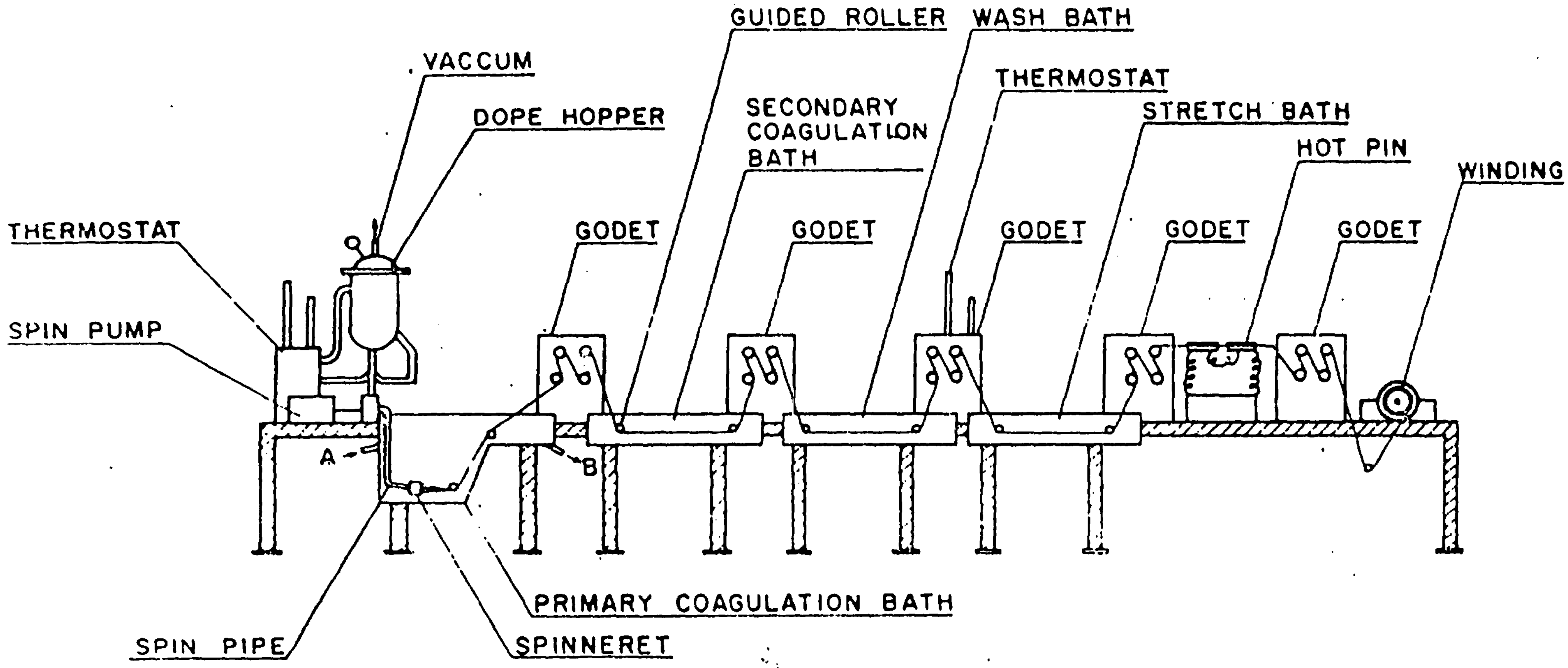
Monomers: acrylonitrile: 94%
Methylacrylate: 6%

ii - Studies in Spinning

With the arrival of Lab. Model Wet Spinning Ass..mply supplied by UNIDO, the activities started in spinning acrylic fibers using the commercial acrylic polymer supplied by FISIBA. The line diagram of the spinning equipment is given in Figure 1. In order to standardize the working condition of this equipment, preliminary spinning experiments were commenced using 23% polymer in N N dimethylformamide (DMF). The results of initial fiber spinning were presented in Table 2. Later, several experiments were made in spinning ing variations in the spin pump speed and successive coagulation, etc.

iii - DSC Studies of the Spun Fibers

With the availability of UNIDO supplied differential scanning calorimeter, it was possible to carry out preliminary studies on CTA spun fibers. The results would through light on the cyclization and stabilization parameters. The DSC results are presented in Table 3.



A,B: COOLING IN-LET AND OUT-LET

FIG. 1 = SPINNIG ASSEMBLEY

TABLE 2 - PRELIMINARY SPINNING RESULTS

SPINNING Nº	STRETCH RATIO	TENSILE STH Kg/mm^2	MODULUS Kg/mm^2	DENSITY G/m^3
1	2.35	7.6	236	1.17
2	2.35	19.4	545	1.17
3	2.25	17.7	600	1.18
7	3.3	14.3	393	1.18
8	3.3	43.3	1297	1.16
9	3.3	35.7	990	1.17
10	3.6	39.3	960	1.16
11	3.6	17.6	408	1.15
12	3.6	20.95	426	1.15
13	3.6	17.4	376	1.16
14	3.5	33.7	937	1.15
15	3.5	33.7	937	1.15
16	5	32.2	415	1.18
17	4	21.4	354	1.19
18	4	22.5	320	1.19
19	4	21.8	466	1.18
20	4	22.1	325	1.17
21	4	21.6	335	1.17
22	4	21.9	362	1.17
23	4	20.2	335	1.17

TABLE 3 - DSC RESULTS OF CTA SPUN ACRYLIC FIBERS

SAMPLE	ORDER REACTION (n)	FREQUENCY FACTOR (min^{-1})	ACTIVATION ENERGY (KJ/MOL)	CORRELATION (%)
1	1,05	6,0 x 10	101,3	99,79
2	1,09	3,0 x 10	99,2	99,53
3	1,04	1,9 x 10	96,7	99,50
7	1,04	8,3 x 10	104,2	99,92
8	1,02	5,4 x 10	102,2	99,75
9	1,06	3,9 x 10	100,5	99,84
10	0,96	3,8 x 10	100,5	99,85
11	1,02	4,6 x 10	100,9	99,92
12	1,01	2,3 x 10	108,7	99,71
13	1,05	1,6 x 10	106,9	99,89
14	1,08	9,7 x 10	104,5	99,80
15	1,05	1,9 x 10	107,8	99,84
16	1,08	2,4 x 10	108,9	99,80
17	1,13	2,8 x 10	109,4	99,58
17A	1,08	8,8 x 10	102,4	99,92
18	1,08	1,2 x 10	105,7	99,87
19	1,12	3,7 x 10	99,8	99,89
20	1,13	1,3 x 10	94,1	99,95
21	1,13	6,5 x 10	102,5	99,92
22	1,12	5,9 x 10	101,9	99,76
22A	1,15	4,3 x 10	100,3	99,84
23	1,15	4,3 x 10	100,3	99,84

2.2- Activities in 1983

i - Activities in Spinning at CTA

Numerous spinning experiments were conducted using Lab. Model Wet Spinning Assembly shown in Figure 1. For example, a few studies are mentioned here.

CASE I

Polymer concentration	: 23% in DMF
Stretch ratio	: 5
Composition of primary coagulation bath	: 50/50 DMF H_2O
Composition of second coagulation bath	: 0-4% DMF in water
Composition of stretch bath	: 6% DMF in water
Composition of wash bath	: water
Temperature coagulation bath	: 1°C
Temperature of second coagulation bath	: 28°C
Temperature of hot plate	: 143°C

CASE II

The temperature of coagulation bath was varied from 0-8°C and the composition of coagulation bath was fixed at 40% DMF in water, stretch ratio was 4. All other conditions were same as in Case I.

In view of the above systematic experiments, Case I and II, the following observations were made:

1- Second coagulation bath (Figure 1) had an observable effect on the physical properties of final fibers.

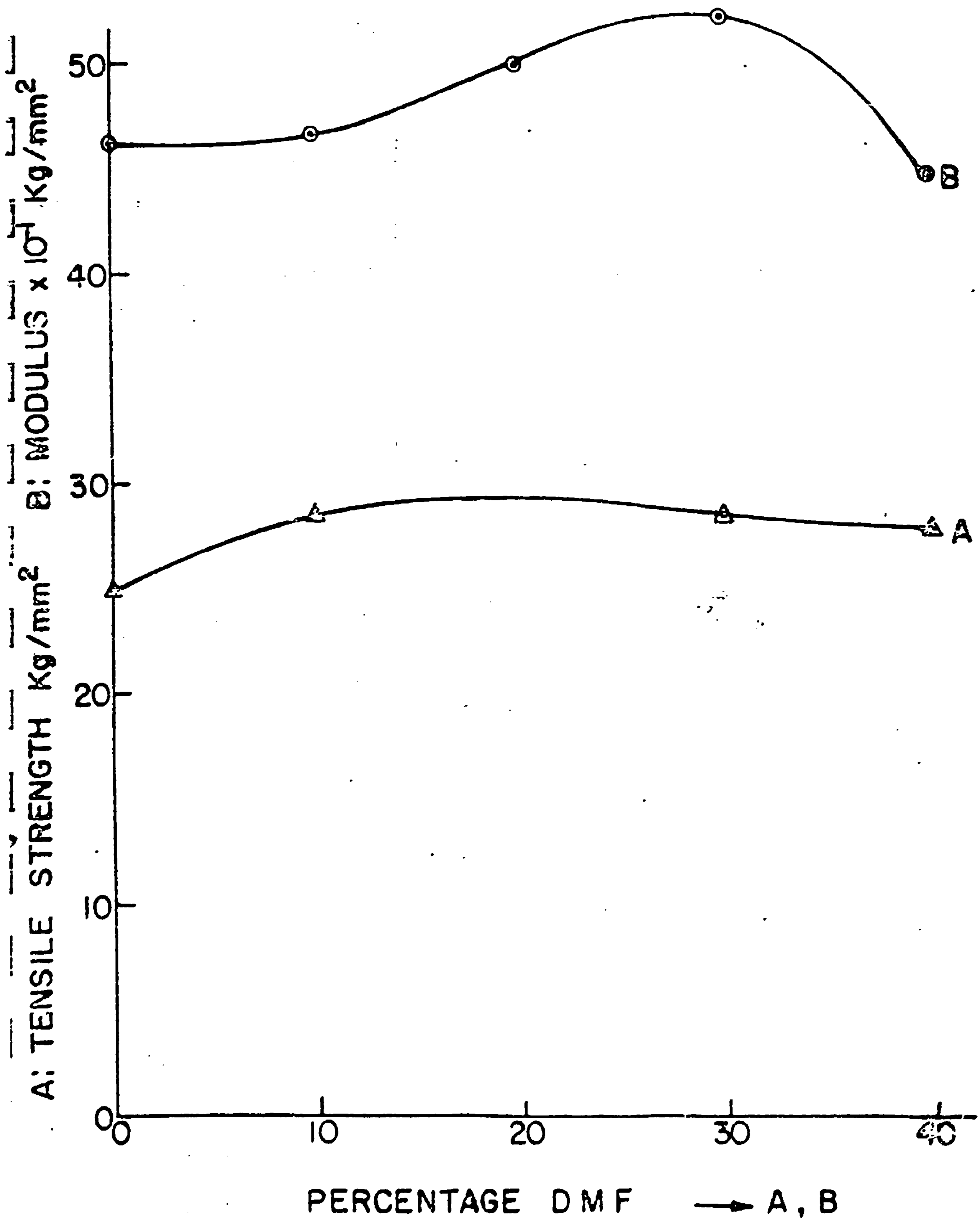
2- Tensile strength of the final fibers found to increase up to a 10% DMF concentration in the 2nd bath (figure 2).

3- Over and above 10% DMF concentration in the 2nd coagulation bath the tensile strength of the fibers was not effected or had minimum effect (Figure 2).

4- Young's modulus found to increase up to about 30% DMF concentration in 2nd coagulation bath (Figure 2).

5- Young's modulus found to decrease when (DMF) was increased above 30% (Figure 2).

6- The increase in the coagulation temperature had decreasing effect on the physical properties of the fiber (Figure 3).



A: TENSILE STRENGTH Kg/mm²   B: MODULUS x 10⁻¹ Kg/mm²
50
40
30
20
10
0
B
A
0
10
20
30
40
PERCENTAGE DMF → A, B

FIGURE -2

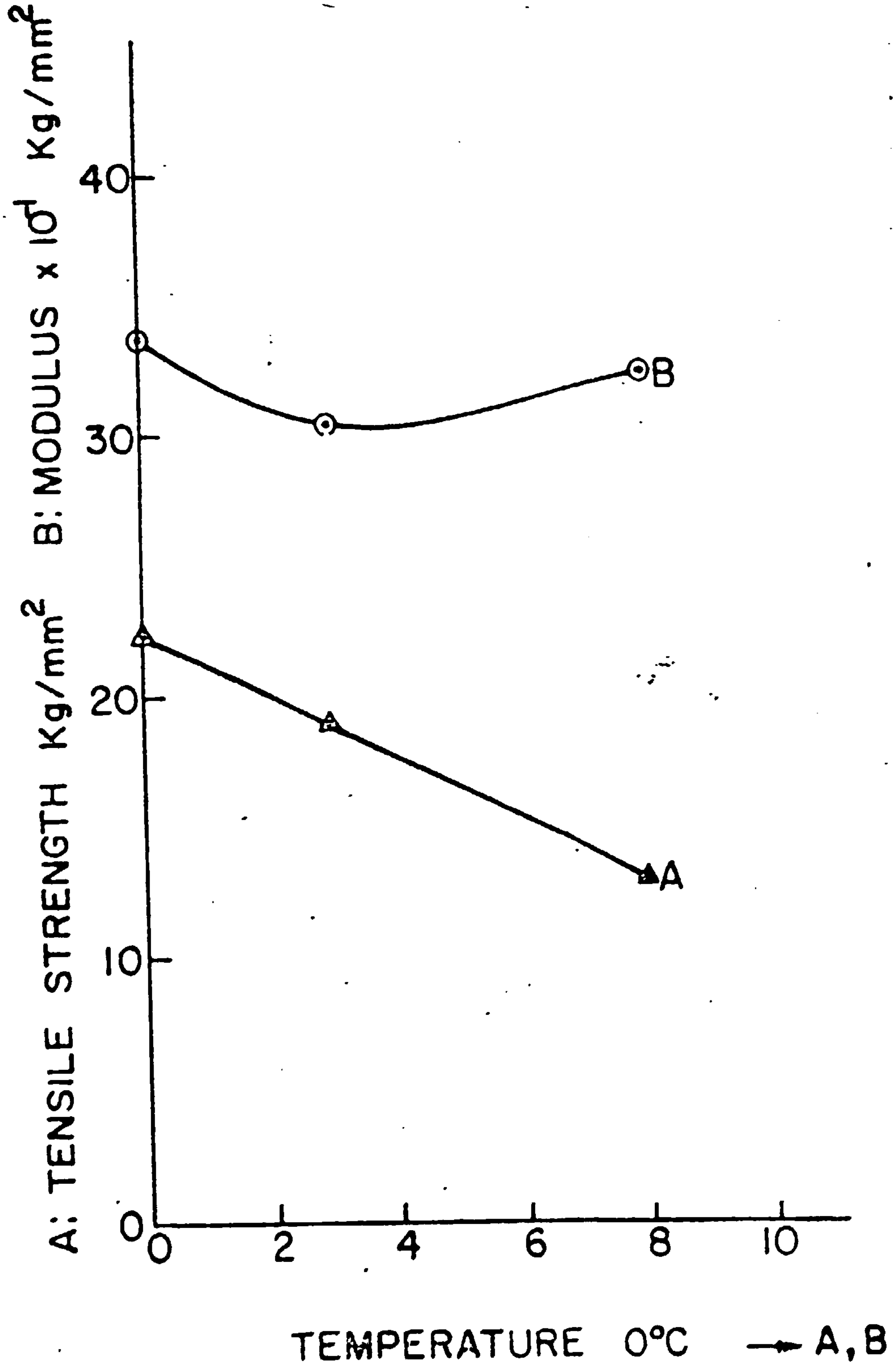

A: TENSILE STRENGTH Kg/mm²   B: MODULUS x 10⁻¹ Kg/mm²
40
30
20
10
0
0
2
4
6
8
10
B
A
TEMPERATURE 0°C → A, B

FIGURE - 3

From these observations, it may be concluded that slow coagulation would yield final fibers with favourable physical properties when the DMF concentration of the 2nd coagulation bath is less than 30% which could be due to the further slow diffision of solvent into the 2nd coagulation bath which would have resulted in the increase of proto fibrile density which in turn would reflect in physical properties of the final fibers. Also it was found lower temperature in the coagulation bath, would also result in high density fibers.

CASE III

Effect of pH of the coagulation bath on physical properties of the acrylic fibers.

Spinning conditions :
Polymer concentration: 24% in DMF
Coagulation bath : 50/50 DMF: H_2O
Spinnerette hole dia.: 0,08mm
Nº of holes : 50
Spin pump temperature: 60°C
Draw ratio : 5.6
Hot spin temperature : 143°C
Composition of stretch bath: 6% DMF, temp.: [illegible]°C
Coagulation bath temperature: Varied
pH of the coagulation bath : Varied

In each spinning experiment, the polymer dope was filtered before introducing into the dope hopper. The dope hopper containing the polymer solution was devaccuated before spinning.

The following observations were made:

- Under the same pH and at different temperatures, the cross section of the final fibers found to change from kidney shape at lower pH values, ie, pHs 2.8, 3.8, 6.6 to circular at higher pH values, ie, pHs 9.1, 10.8.
- However, under the same temperature and in different pH values, there was not much effect on the cross section, showing that pH may not effect on the structural aspects of the fibers. However, in certain cases at high pHs voids are noticed in the optical microscopy.
- Density decreased with an increase in the pH of the coagulation

bath, thereby effecting the mechanical properties of the final fibers.

- The increase in pH values found to have diminishing effect in general on tensile strength values of the fibers. At coagulation temperatures of 4°C, 15°C, 20°C, 25°C a decreasing trend in general was observed. However, there are some discrepancies in some places. Overall picture is that there is a decreasing effect.

- Modulus values are also in general decreased with increasing pH, however, a decrease and increase and decreasing tendencies were observed. The general trend, however, is of decreasing order. These decreasing trends in tensile strength and modulus would have resulted due to decreasing values of density with increasing pH.

CASE IV

Effect of spin pump speed, size and speed of take-up godet on physical properties of acrylic fibers

In wet spinning, the denier of the fiber depends on several spinning parameters such as, spin pump speed, spin pump size (that controle the pumping of polymer dope into the coagulation bath), spinnerette hole diameter, number of spinning holes, take-up speed and hot stretch conditions. While keeping all conditions constant, the fineness of the fiber could be improved by increasing the number of holes of the spinnerette. In cases where there are no spinnerettes with a higher number of holes (in our cases we have only 100 holes spinnerette), the fineness can be improved by reducing the spin pump speed there by controlling the polymer pumping into the coagulation bath. When this is also achieved maximum the next parameter that can be used to get the fine fibers is by reducing the spin pump size. However, when the minimum size pump has already been used, then the take-up speed of the first godet could be increased which facilitates to increase the speed of the subsequent godet. This will not only give fine fibers, but also would increase the speed of production. However, when doing so, one may sacrifice the density of the fiber unless the length of coagulation path is increased. Eventhough various reports are available in literature systematic studies on these parameters are lacking and also most of the results are in patent form. In view of this, a systematic study is conducted using different spin pumps, spin pump speeds, take up roller speeds under identical conditions.

Spinning conditions in Case IV were:

Polymer concentration	: 23% in DMF
Viscosity of spinning solution at 50°C by ball fall method	: time: 23
Coagulation bath composition	: 50/50 DMF H_2O
Spinnerette holes	: 100
Spinnerette dia.	: 0,1 mm
Composition of wash bath	: Baths 1 and 3 water
Composition of stretch bath	: 6% DMF
Temperature of coagulation bath	: 2-3°C
Temperature of hot pin	: 170°C

Maintaing the above spinning conditions, in general, the other variables were changed as given in Table 4.

TABLE 4 - SPINNING RESULTS

	SPIN PUMP SPEED METERS/MINUTE	SPEED OF TAKE UP GODET 1 METERS/MINUTE	MAXIMUM STRETCH OBTAINED	DENIER OF THE FIBER
Spin pump size .6cm^3/Rev.	3.0	5	7.0	3.990
	2.0	5	7.0	2.470
	1.4	5	6.4	1.828
	1.4	7	6.7	1.560
Spin pump size .4cm^3/Rev.	1.4	5	6.2	1.430
	1.4	6	5.4	1.370
	1.4	7	5.7	1.009
	1.4	7	6.0	1.020
	1.4	7	6.3	0.970
	1.4	8	5.6	0.960

Using spin pump of size 0,6 cm^3/Rev when the speed of the spin pump is 3M/m a stretch ratio of 7 could be obtained which resulted in a denier of 3.99. When the spin pump is further reduced to 2M/m a stretch ratio of 7 is achieved (difficult), but the denier of the fiber reduced. A further reduction resulted in a decrease of stretch ratio but denier of the fiber reduced to 1.828. This shows that a decrease in rate of

TABLE 5 - BEST SPINNING RESULTS OF 1983 (LAB. MODEL)

	STRETCH RATIO	DENIER	TENSILE STRENGTH GPa	MODULUS GPa
EARLY 1983	5.6	3.79	0.36	5.8
	5.6	8.20	0.38	5.4
	5.6	1.92	0.34	7.8
2nd HALF OF 1983	4.9	1.35	0.28	7.72
	5.7	1.00	0.34	9.24
	6.0	1.02	0.28	8.04
	6.3	0.97	0.421	7.73
	5.6	0.94	0.349	9.86

pumping of polymer into the coagulation would yield finer fiber keeping al other spinning conditions constant. However, the reduction in the speed of the pump will impose a constrain in the maximum obtainable stretch ratio keeping stretch bath conditions constant. Beyond these limits the fibers were breaking in the stretch bath. However, by increasing the take-up godet to 5m/m to 7m/m a slight raise in stretch ratio and denier could be achieved.

When the pump size was further decreased from 0.6 to 0.4 cm^3/Rev. still finer fibers could be obtained. As shown in Table 4, when the take-up godet speed is changed, very fine fibers could be obtained.

However, the rise in the speed of take-up godet would impose some restriction in spinning. A quicker removal of the fiber from the coagulation bath might effect the density of the fiber which may in turn result in weaker fibers. In such case, one should compensate by increasing the coagulation path.

On the basis of these experiments, the following conclusions were made:

- A decrease in size of the spin pump would yield fine fiber due to the reduction in the pumping of polymer into the coagulation bath.
- Decrease in spin pump speed would also yield finer fibers due to the above reason.
- Care should be taken to compensate the residence time of proto fiber in the coagulation bath by increasing spinning path.
- The reduction in spin pump size and spin pump speed would impose restrictions on obtainable draw ratio.

Later on hundreds of experiments were conducted. The variations in spinning parameters included spin pump speeds, no of holes in the spinnerette, size of the spinnerette hole, dope composition, etc. The best spinning results of 1983 are represented in Table 5. The results during the later half of the year are comparable with some of commercial fibers.

ii - <u>Spinning Activities at FISIBA</u> (Fibras Sintéticas da Bahia S.A., Camaçari, Bahia)

Meanwhile the pilot plant spinning assembly supplied by UNIDO has arrived. This was assembled at the previously agreed site FISIBA. The visit of Prof. Falkai was planned at the final stages of the commissioning of this equipment. Myself and Dr Falkai visited again the FISIBA, and the pilot plant spinning assembly site and rectified the small defects and started the initial spinning experiments. The FISIBA scientists were trained in several of the spinning experiments. FISIBA, though a fiber producing industry had no previous wet spinning experience They were trained in understanding the spinning conditions and crucial points. The preliminary spinning results at FISIBA towards the end of 1983 are presented in Table 6. After the preliminary testing in FISIBA at the plant site remaining tests like density measurements were performed at CTA. In view of the fact that the density of the fiber was less it was adviced to them to perform a set experiments consisting the variations in polymer composition. The experiences in the bench scale experiments at FISIBA enabled to identity and to improve the properties of fibers in the pilot plant. Also, the scientists trained at CTA by me and Falkai further helped the FISIBA people in commissioning the preliminary spinning experiments. In view of mutual contacts between CTA scientists and FISIBA people, the properties of the fibers at the pilot plant were improved. These contacts and the interest shown by FISIBA were slowly growing and FISIBA and its parent organization COPENE - Petroquímica do Nordeste S.A. were understanding the potentials of carbon fibers. This is very good for a project of this type for further industrialization of this technology in the country.

TABLE 6 - PRELIMINARY SPINNING RESULTS AT PILOT PLANT IN FISIBA

Spinning nº	Stretch	Final Denier	TS GPa	Modulus GPa	Density
1	4	5.77	2.22	-	-
2	2.99	3.1	1.616×10^{-1}	4.31	1.43
3	3	2.2	2.449×10^{-1}	5.292	1.14
4	4.11	1.9	2.711×10^{-1}	5.542	1.14
5	4.47	1.9	3.163×10^{-1}	6.119	1.13
6	4.94	1.84	3.21×10^{-1}	5.36	1.18

2.3 - Activities in 1984

i - Spinning Activities at CTA

In order to conduct the preliminary heat treatment experiments on CTA spun fibers, the fibers were spun with selected best conditions of 1983 in larger quantities. Single spinnings were conducted for 7-8 hours with lab spinning assembly at CTA. This time constrain is because of the dope container capacity. However, the fibers were thus spun for prolonged times in a number of times keeping the spinning parameters constant. The spinnerette used at CTA had only a minimum of 100 holes. Thus the fibers from a number of bobbins were pooled together. To make this, the precursor group constructed an equipment. These fibers were pooled into a tow consisting of at least 1000 filaments and preliminary heat treatment experiments were conducted at CTA. Upon the analysis of preliminar results on the heat treatment of the CTA spun fibers, it was felt necessary:

- To increase the density of the fiber
- To further stretch the fibers while spinning.

In view of these observations, a set of spinning experiments were designed by me to increase the density of the fiber and to heat stretch the fiber in spinning. These included the spinning with increased dope concentrations and increase path length of coagulation bath.

ii - Spinning Activities in FISIBA

Upon my advice to change the spinning conditions such as dope concentration, etc., FISIBA people have spun fibers again. The spun fibers were sent to CTA. The results of the fibers are given below in Table 7.

TABLE 7 - SPINNING RESULTS AT FISIBA IN 1984

SPINNING Nº	FINAL DENIER	TS GPa x 10^{-1}	MODULUS GPa	DENSITY
7	1.64	3.53	6.258	1.19
	1.71	8.19	5.75	1.15

iii - Studies in Polymerizations

Meanwhile the UNIDO supplied polymerization reactors chemical reagents, initiators and monomers arrived at CTA, polymerization experiments were conducted in smaller scale initially using two monomers, ie acrylonitrile and methylacrylate, and acrylonitrile, methylacrylate and itaconic acid. The smaller scale polymerizations were of 1 litre capacity. These polymerizations were conducted in aquous medium. The polymeris were filtered, dried and characterized. The results are given in Tables 8 and 9.

TABLE 8 - RESULTS OF POLYMERIZATION CONDUCTED IN 1 LITER CAPACITY USING TWO MONOMERS, ie ACRYLONITRILE AND METHYLACRYLATE

EXPERIMENTS	AN (%)	MA (%)	TEMPER. (°C)	TIME (h)	QUANT. (g)	YIELD (%)
A - 1	97	3	40	5	104,70	52,35
2	97	3	35	5	84,91	42,455
3	97	3	40	5	171,52	85,76
4	97	3	50	5	171,50	85,75
5	97	3	60	5	173,94	86,97
6	97	3	70	5	164,71	82,355
7	97	3	80	5	172,00	86,00
B - 8	96	4	40	5	71,71	35,855
9	96	4	60	5	109,77	54,885
10	96	4	50	5	157,72	78,86
11	96	4	70	5	185,24	92,62
12	96	4	80	5	179,11	89,555
C- 13	95	5	40	5	29,95	14,975
14	95	5	40	5	58,73	29,365
15	95	5	50	5	26,84	13,42
16	95	5	70	5	142,40	71,20
17	95	5	80	5	165,10	82,55
18	95	5	60	5	83,56	41,78
D- 19	98	2	40	5	53,57	26,785
20	98	2	50	5		
21	98	2	60	5		
22	98	2	70	5		
23	98	2	80	5		
E- 24	99	1	50	5		
25	99	1	70	5		
26	99	1	40	5		
27	99	1	60	5		
28	99	1	80	5		

TABLE 9 - RESULTS OF POLYMERIZATION CONDUCTED IN 1 LITER CAPACITY USING THREE MONOMERS - ACRYLONITRILA (AN), METHYL ACRYLATE (MA) AND ITACONIC ACID (ITA)

EXPERIMENTS	AN (%)	MA (%)	ITA (%)	TEMPER. (°C)	TIME (h)	QUANT. (g)	YIELD (%)
A - 29	96	3	1	40	5		
30	96	3	1	60	5		
31	96	3	1	70	5		
32	96	3	1	50	5		
33	96	3	1	80	5		
B - 34	95	4	1	50	5		
35	95	4	1	60	5		
36	95	4	1	70	5		
37	95	4	1	40	5		
38	95	4	1	80	5		
39	95	4	1	80	5		
C - 40	94	5	1	70	5		
41	94	5	1	60	5		
42	94	5	1	80	5		
43	94	5	1	40	5		
44	94	5	1	50	5		
D - 45	94	4	2	50	5		
46	94	4	2	40	5		
47	94	4	2	60	5		
48	94	4	2	70	5		
49	94	4	2	80	5		
E - 50	94	3	2	40	5		
51	94	3	2	60	5		
52	94	3	2	50	5		
53	94	3	2	70	5		
54	94	3	2	80	5		
F - 55	95	3	2	40	5		
56	95	3	2	50	5		
57	95	3	2	60			
58	95	3	2	70	5		
59	95	3	2	80	5		

iv - Planning in Polymerizations

Also several of the polymerizations were planned and the following plan was given to the precursor group:

Planned Polymerization Experiments:

- Polymerization in one liter capacity	- variation in temperature
- Polymerization in one liter capacity	- constant temperature
- Polymerization of acrylonitrile in the presence of two comonomers	- variation in temperature (third monomer)
- Polymerization of acrylonitrile in the presence of two comonomers	- constant temperature
- Polymerization of acrylonitrile in the presence of two comonomers	- variations in temperature (third monomer)
- Polymerization of acrylonitrile in the presence of two comonomers	- constant temperature (third monomer)
- Characterization of these copolymers	

v - Scaling up of Polymerization to Higher Capacity

A - Scale up of acrylic polymerizations (using monomers, acrylonitrile and methylacrylate) to 5 liter capacity should be continued.

B - Scale up of acrylic polymerizations (using monomers, acrylonitrile, methylacrylate and itaconic acid) should be continued after the analysis of the preliminary results.

2.4- Additional Spinning Studies

i - Spinning of CTA Acrylic Polymers

In future, the best of CTA polymers have to be spun using the previous and modified spinning parameters. The group was advised by me on this. I have also advised the group to prepare polymers of type B in 10 liter capacity after looking at the initially spun A and B CTA polymers. These polymers have to spun using the previously developed spinning conditions.

ii - Planning in New Spinning Methods

Spinning of acrylics polymers using inorganic spinning solvents:

I have advised and gave the planned sheets of the type enclosed to spin the acrylic fibers using inorganic solvents. Inorganic solvents would permit in using high spinning temperature in the coagulation bath which would result in circularity in cross section of spun fibers. These inorganic solvents could be sodium thiocynate and zinc chloside. Representative planned sheets are enclosed as Tables 10 and 11.

SPINNING VARIABLE

DATE :

POLYM.CONC.: Different polymer concentrations, ie 10-30% have to be used in 55% NaScN

COMPO.COAG.BATH: Diff.solvent/H_2O compositions have to tried (10 to 30% NaScN)

SPINNERET HOLE DIA.: Available spinneretts have to be used

SPINNERET Nº OF ROLES: Spinnerette with higher spinnerette holes to be used

POLYMER BATCH Nº: use CTA polymer

VISCOSITY: to be determined every line

AT TEMP. :

Conditions / Batch nº	Temp Spin Bloc °C	Spin Pump Size	Spin Pump RPM m/min	Speeds of G1 m/min	Speeds of G2 m/min	Speeds of G3 m/min	Speeds of G4 m/min	Speeds of G5 m/min	Temp. Coag. Bath °C	Wash Baths 1 Temp. °C Comp.	Wash Baths 2 Temp. °C Comp.	Wash Baths 3 Temp. °C Comp.	St. Bath Temp. °C	St. Bath Comp.	St. Ratio	Hot plate °C	Denier Wet	Denier Dry	Cross Area	Density g/cm^3	Cross Sec.	% DMF in fibre	TS kgf/mm²	M kgf/mm²	Order (m)	Freq. factor (min^{-1})	[illegible] (KJ/MOL)	Remarks
Use best of previous spinning results and modify as and when required as per the system used.																												

TABLE 11 – PLANNING OF SPINNING USING ZINCCHLORIDE.

NOTE: Detail sheet were given to the Precursor Group

SPINNING VARIABLE

DATE :

POLYM.CONC.: Use 10 to 25% polymer compositions and arrive opt.compsitions

COMPO.COAG.BATH: Use 20 to 50% $ZnCl_2$ concentration and arrive at good composition

SPINNERET HOLE DIA.: Use available spinnerettes

SPINNERET Nº OF ROLES: Use spinnerette with maximum nº of holes

POLYMER BATCH Nº: Use CTA polymer

VISCOSITY:

AT TEMP. :

Conditions / Batch nº	Temp Spin Bloc °C	Spin Pump: Size	Spin Pump: RPM m/min	Speeds of: G1 m/min	Speeds of: G2 m/min	Speeds of: G3 m/min	Speeds of: G4 m/min	Speeds of: G5 m/min	Temp. Coag. Bath °C	Wash Baths 1: Temp. °C Comp.	Wash Baths 2: Temp. °C Comp.	Wash Baths 3: Temp. °C Comp.	St. Bath: Temp. °C	St. Bath: Comp.	St. Ratio	Hot plate °C	Denier: Wet	Denier: Dry	Cross Area	Density g/cm^3	Cross Sec.	% DMF in fibre	TS kgf/mm²	M kgf/mm²	Order (n)	Freq. Factor (min⁻¹)	[illegible] (...OL)	Remark
Use best of previous spinning results and modify wherever and whenever required.																												

3.0 - Development Activities in Heat Treatment

3.1- Activities in 1982

i - Position at the Time of my Arrival

Just before my arrival, the previous Chief Technical Adviser, Dr O.P.Bahl left the Project. At Centro Técnico Aeroespacial, the heat treatment activities were abandoned on FISIBA commercial grade fibers, the research and developmental activities started already using imported special grade acrylic fibers supplied by Courtaulds, England. At this time, the developmental activities on heat treatment were conducted using the heat treatment system of the type shown in Figure 4. The best results on FISIBA fibers at the end of 1981 are shown in Table 12.

ii - Activities After my Arrival

Several studies were conducted in 1982. Several of the parameters in heat treatments were changed due to the advices of other experts who visited the project site. The studies were concentrated mainly using Courtauld's SAF precursor. Several of experiments were conducted in heat treatment.

Tables 13 and 14 and figures 5 and 6 show some of the initial studies on Courtauld's fibers on the effect of heat treatment temperature on tensile strength and modulus. The best results of heat treatment studies in 1982 are represented in Table 14A (continuous process) and table 14B (discontinuous process). Also, in order to find out the effect of air flux during oxidation stage, several experiments were conducted both in the presence and absecne of moved air. The results are presented in Table 15.

FIGURE 4 - I OLD SYSTEM

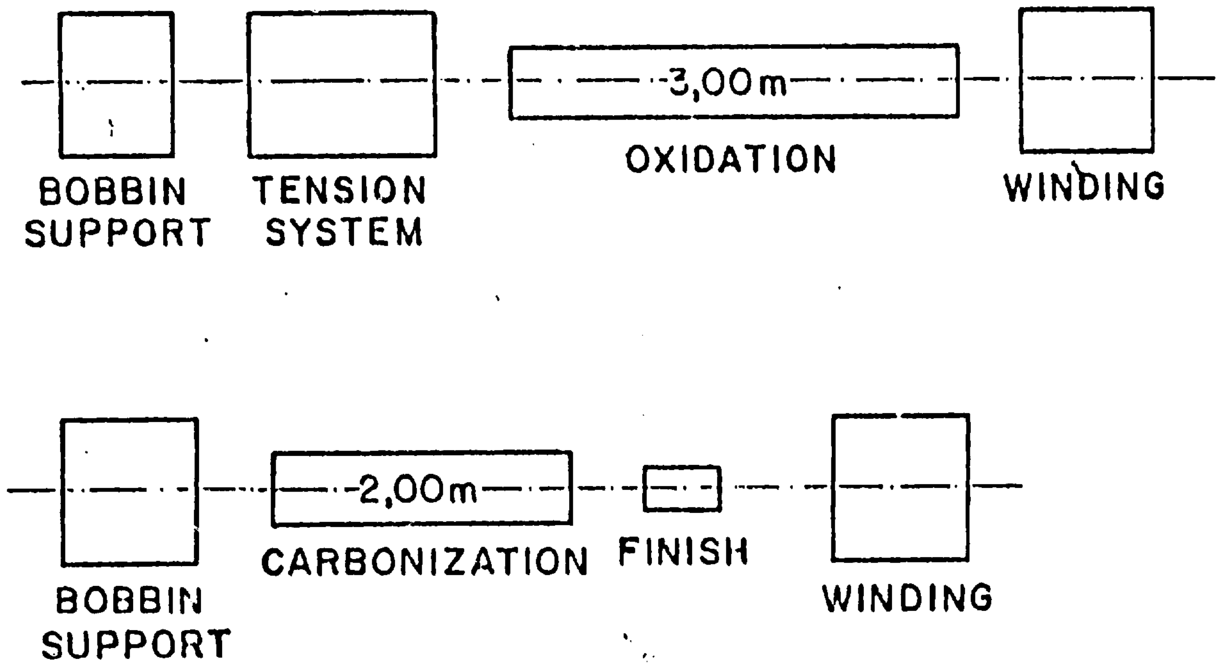


TABLE 12 - BEST RESULTS OF 1981 - ONE STEP OXIDATION (FISIBA PRECURSOR)

	OXIDATION				CARBONIZATION			CARBON FIBERS PROPERTIES	
	ELONGATION %	TEMPERATURE °C	RES.TIME MIN.	ATMOSPHERE	TEMPERATURE °C	RES.TIME MIN.	ATMOSPHERE	T.S.	MODULUS
CONTINUOUS PROCESS	+7	230	180	AIR	1000	120	ARGON	200×10^3 PSi 1,38 GPa	20×10^6 PSi 138 GPa
DISCONT. PROCESS.	+6	220	220	FORCED AIR	1000	120	ARGON	345×10^3 PSi 2,30 GPa	26×10^6 PSi 180 GPa

TABLE 13 - EFFECT OF CARBONIZATION TEMPERATURE

CARBON TEMP. °C	DENSITY g/cm^3	CROSS AREA 10^{-5} mm^2	TENSILE STRENGTH (GPa)	TENSILE MODULUS (GPa)
1000	1,80	37,3	1,7 $\pm$ 0,5	180 $\pm$ 27
1100	1,76	36,9	2,1 $\pm$ 0,6	190 $\pm$ 22
1200	1,76	37,1	2,2 $\pm$ 0,5	180 $\pm$ 23
1300	1,75	36,3	2,6 $\pm$ 0,7	200 $\pm$ 25
1400	1,75	35,4	2,7 $\pm$ 0,6	212 $\pm$ 31
1500	1,75	34,8	2,1 $\pm$ 0,6	215 $\pm$ 29
1600	1,77	35,3	1,6 $\pm$ 0,4	215 $\pm$ 23
1800	1,77	35,5	1,6 $\pm$ 0,5	216 $\pm$ 23
2000	1,79	34,5	1,5 $\pm$ 0,4	245 $\pm$ 29
2250	1,81	34,8	1,8 $\pm$ 0,4	222 $\pm$ 36
2500	1,86	33,5	1,9 $\pm$ 0,5	279 $\pm$ 36

TABLE 14 - EFFECT OF STRETCH DURING CARBONIZATION

CARBON TEMP. °C	STRETCH %	DENSITY g/cm³	CROSS AREA 10^{-5} mm²	TENSILE STRENGTH (GPa)	TENSILE MODULUS (GPa)
1000	0	1,80	38,4	2,1 ± 0,4	176 ± 30
1000	5	1,78	35,4	2,2 ± 0,4	199 ± 35
1000	10	1,77	35,1	2,5 ± 0,3	203 ± 19
1000	15	1,78	32,9	2,8 ± 0,4	210 ± 25

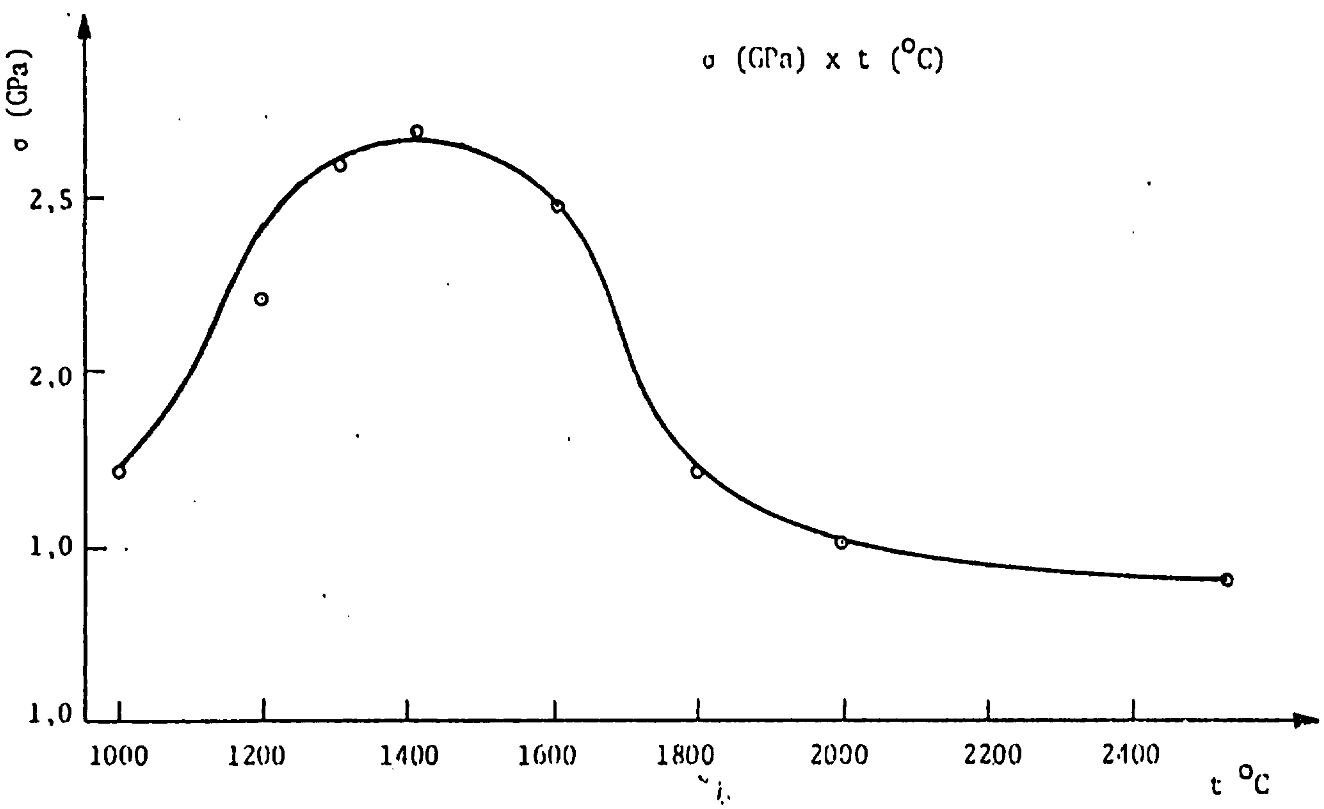

σ (GPa) x t (°C)
σ (GPa)
2,5
2,0
1,0
1,0
1000
1200
1400
1600
1800
2000
2200
2400
t °C

FIGURA 5 -

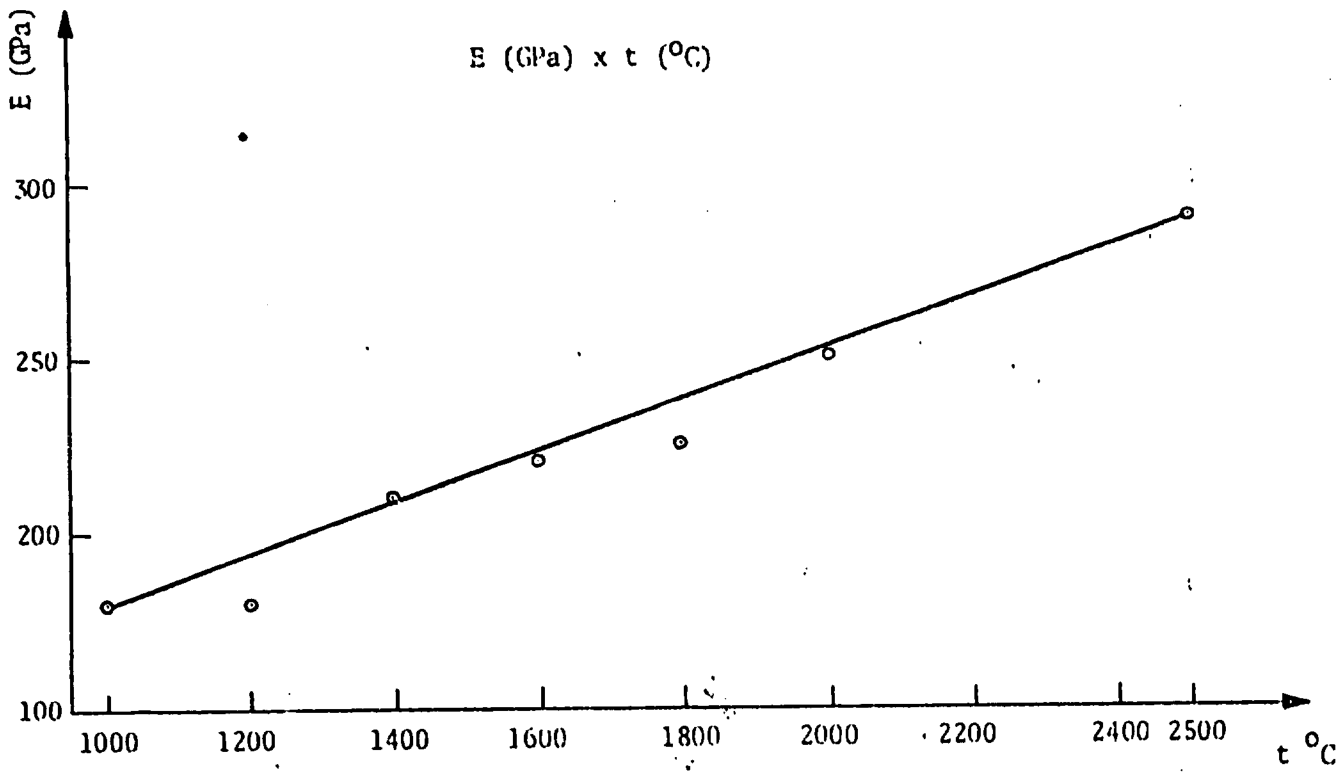

E (GPa)
E (GPa) x t (°C)
300
250
200
100
1000
1200
1400
1600
1800
2000
2200
2400
2500
t °C

FIGURA 6 -

TABLE 14A- BEST RESULTS OF 1982 (COURTAULDS SAF PAN)

CONTINUOUS PROCESS

ELONGATION PRE-STRETCH %	TEMPERATURE PRE-STRETCH °C	PRE-OXID. TEMP. °C	OXIDATION TEMP. °C	RES.TIME PRE-OXID. MIN.	RES.TIME OXID. MIN.	ATM.	TEMP. CARB. * °C	RES.TIME CARB. MIN.	ATM. CARB.	T.S.	MODULUS
+6	190	220	230	60	120	AIR	1100	120	ARGON	275×10^3 PSi 1,90 GPa	21×10^6 PSi 145 GPa

* SHRINKAGE = (8-9)%

TABLE 14B - BEST RESULTS OF 1982 (COURTAULDS SAF PAN)

DISCONTINUOUS PROCESS

ELONGATION OXID. %	TEMPERATURE OXID. °C	RES.TIME MIN.	ATMOSPHERE OXID.	TEMPERATURE CARB. * °C	RES.TIME MIN.	ATMOSPHERE CARB.	T.S.	MODULUS
+6	220	200	FORCED AIR	1100	120	ARGON	350×10^3 PSi 240 GPa	$2,8\times10^6$ PSi 193 GPa

* SHRINKAGE = (8-9)%

TABLE 15 - EFFECT OF AIR FLUX ON THE PHYSICAL PROPERTIES OF CARBON FIBERS

Stretching Ratio (%)	190°C Density (g/cm³)	190°C Weight/m (mg)	230°C Density (g/cm³)	230°C Weight/m (mg)	265°C Density (g/cm³)	265°C Weight/m (mg)	1000°C Density (g/cm³)	1000°C Weight/m (mg)	1000°C Cross Area (μm²)	1000°C Tensile Strength (GPa)	1000°C Tensile Modulus (GPa)
WITHOUT AIR FLUX											
-5	1,21	779	1,35	806	1,43	795	1,77	451	42,5	2,4 ± 0,7	199 ± 32
0	1,21	711	1,35	749	1,43	755	1,77	422	39,7	2,0 ± 0,6	204 ± 28
+5	1,21	690	1,35	698	1,43	725	1,77	404	38,0	2,2 ± 0,7	214 ± 52
+10	1,21	668	1,35	693	1,43	707	1,77	400	37,7	2,8 ± 0,4	220 ± 30
WITH FLUX OF 35L AIR/MIN.											
-5	1,21	765	1,32	816	1,46	793	1,75	450	42,8	2,1 ± 0,4	166 ± 16
0	1,21	740	1,30	758	1,46	780	1,75	438	41,7	1,8 ± 0,5	156 ± 25
+5	1,21	714	1,30	714	1,46	727	1,75	413	39,3	2,0 ± 0,6	188 ± 28
+10	1,21	673	1,30	674	1,46	693	1,75	405	38,6	2,7 ± 0,5	197 ± 19

3.2- Activities in 1983

In view of the so-far gained experience, a few modifications were effected in the design of the existing lab. model processing equipment. For easy handling and temperature control, the existing stabilization furnace was cut into two. UNIDO supplied trios were introduced in between the furnaces for easy controlling of the movement of the fiber during processing. The modified versions are shown in Fig.7 and 7A which also include the winding equipment supplied by UNIDO.

Meanwhile CTA/PMR has built a new building for the carbon fiber group, the oxidation and carbonization processing equipment was assembled in line for the continuous processing in the new building.

In view of the several of the experiments conducted by the group and due to availability of useful results, I inspired the group to present the results in the American Carbon Society in 1983 in San Diego. The group and myself identified the subject and more vigorous studies were conducted on these lines. Also the expert Th.Mueller was very much helpful in this venture. Prof. Fitzer had also encouraged the group. This is for the first time the CTA/PMR Carbon Fiber Group presenting results in an outside Conference. In view of these effects, a paper (Annex I) was presented. The results were appreciated by the participants in the Conference.

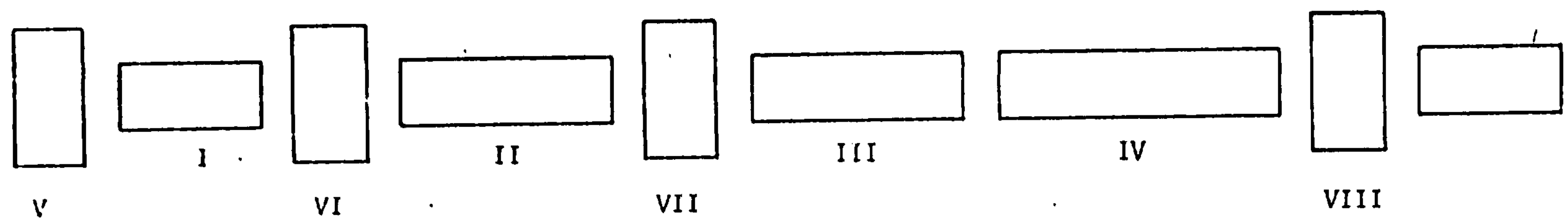
3.3- Activities in 1984

i - Studies in Heat Treatment

As per the advice of the expert Dr Kalnin, experiments were conducted by modifying the processing conditions. The main aspect of these studies were modified stabilization temperatures. Stabilization studies were conducted at three stabilization temperatures, ie, 220-230°C, 250-270°C and 270-300°C. From these studies it was observed that closer the individual furnaces, better properties were obtained. DSC experiments were conducted at every stage of heat treatment. Some of the results using three stabilization experiments are presented in Table 16 and Figures 8 and 9.

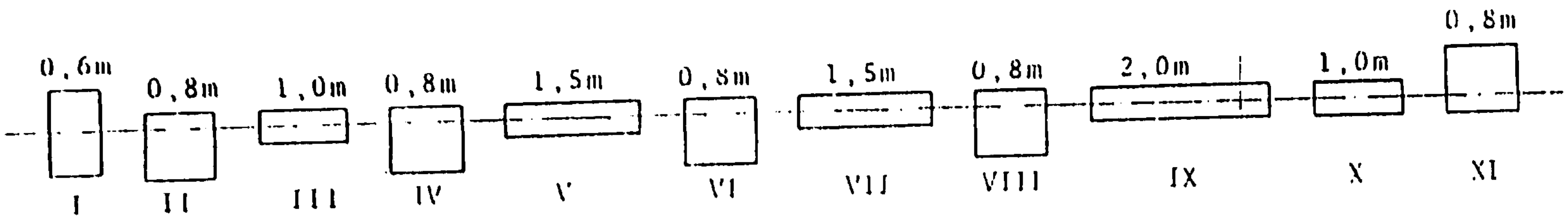
In order to explore the additional oxidants, studies were conducted

FIGURE 7 - LINE DIAGRAM OF PROCESSING EQUIPMENT USED FOR RESULTS IN TABLE 16, FIGURES 8, 9.



I, II, III - Stabilization furnaces
IV - Carbonization furnaces
V, VI, VII, VIII - Trios
IX - Bobbin

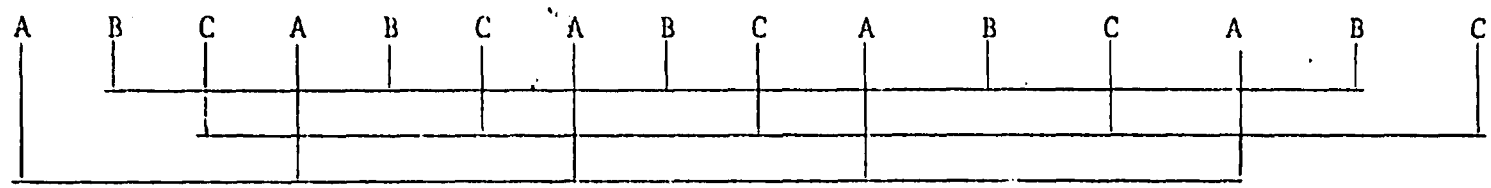
FIGURE 7A - NEW INSTALLATION OF CARBON FIBER PROCESSING EQUIPMENT



I - P.A.N.
II - Trio nº 1
III - pre-stretching
IV - trio nº 2
V - stabilization furnace nº 1
VI - trio nº 3
VII - stabilization furnace nº 2
VIII- trio nº 4
IX - pre-carbonization furnace
X - carbonization furnace
XI - winding

TABLE 16 - RESULTS OF CARBONIZATION USING 3 STAGES STABILIZATION

	I			STEP II			STEP III			STEP IV					
										TOGETHER			SEPARETED		
Temperature °C	200_A	200_B	240_C	230_A	230_B	280_C	265_A	280_B	300_C	1.100_A	1.100_B	1.100_C	1.100_A	1.100_B	1.100_C
Linear density (mg/m)	676	676	723	708	708	732	718	704	697	382	351	350	371	344	356
Volume density (g/cm^3)	1,22	1,22	1,36	1,38	1,38	1,46	1,45	1,48	1,51	1,14	1,72	1,72	1,72	1,70	1,76
DSC Test (°C)	217	217	245	255	255	310	*	*	*	-	-	-	-	-	-
tm (°C)	308	308	328	343	343	350	*	*	*	-	-	-	-	-	-
Δh (°C)	2,79	2,79	0,763	0,550	0,550	0,210	*	*	*	-	-	-	-	-	-
Young's modulus (GPa)	10,6	10,6	7,2	8,34	8,34	6,47	9,32	9,12	9,05	220	197	192	214	184	187
Tensile strength (GPa)	0,58	0,58	0,31	0,35	0,35	0,21	0,24	0,22	0,21	2,58	2,47	2,39	2,30	2,22	2,31



* Complete

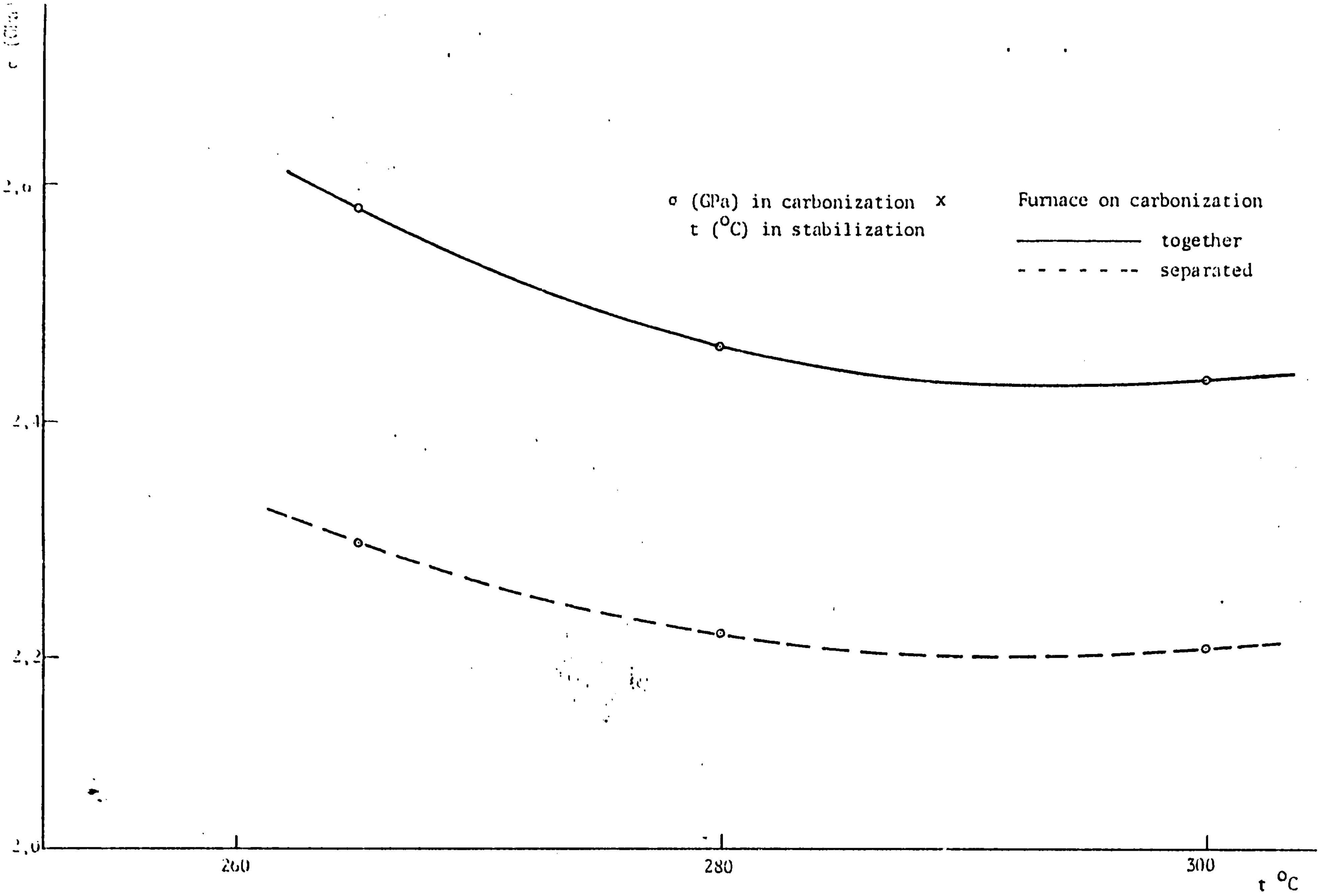

σ (GPa) in carbonization x
t (°C) in stabilization
Furnace on carbonization
together
separated
2,1
2,2
2,0
260
280
300
t °C
Stabilizatic

FIGURE 8

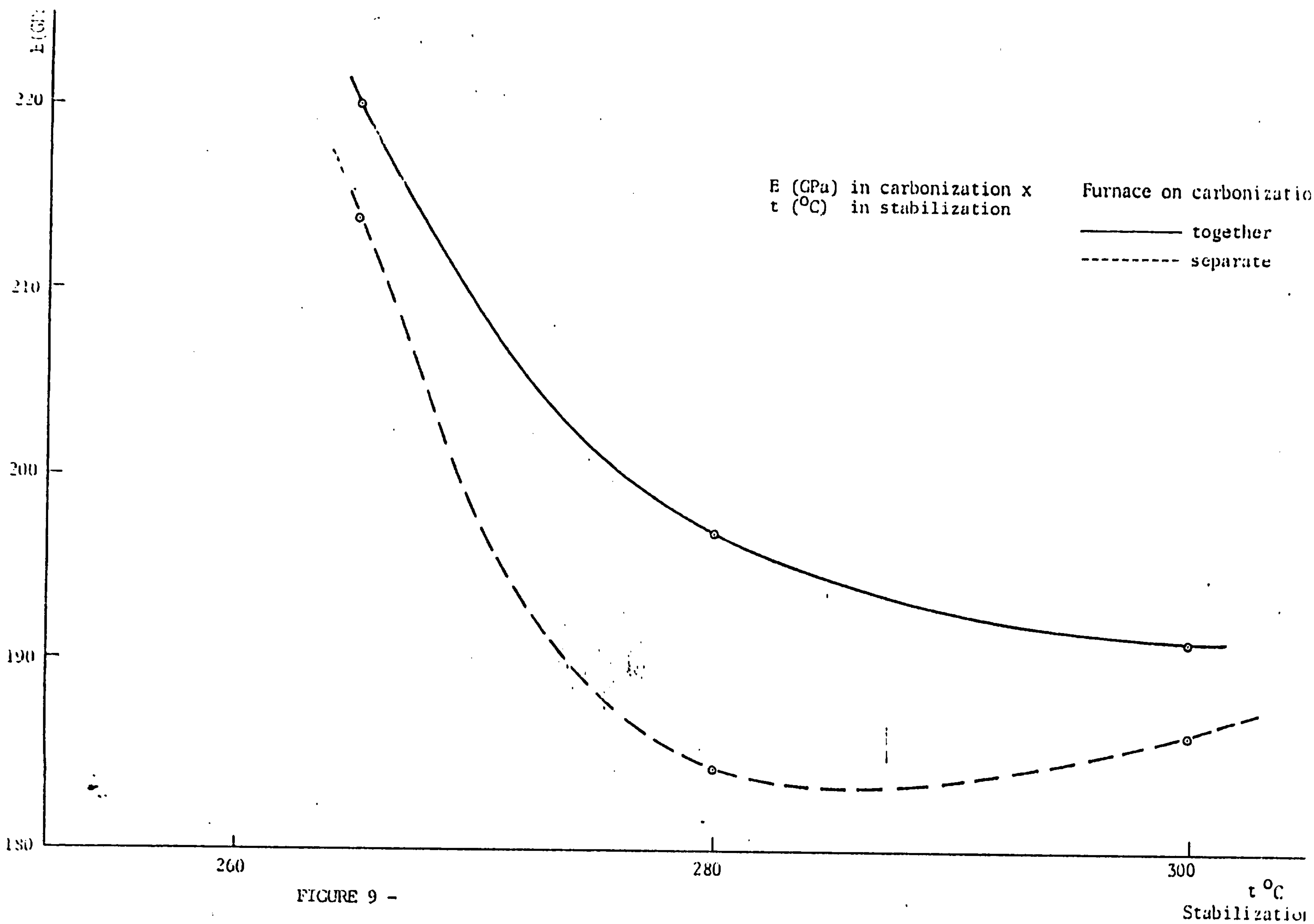

E (GPa)
220
210
200
190
180
260
280
300
t °C
Stabilizatio
E (GPa) in carbonization x
t (°C) in stabilization
Furnace on carbonizatio
together
separate

FIGURE 9 -

using NO_2 as the oxidants. It was observed that NO_2 stabilization did not improve the physical properties. These results were presented in Carbon 84 Conference in France (Annex II).

ii - Activities in Oxidized Polyacrylonitrile (PAN-Ox)

PAN-Ox is a good substitute for Asbestos. It is a heat resistant material and has several of industrial applications. Some of the companies which participated in the Conference at CTA were interested to use oxidized polyacrylonitrile fiber as thermally resistant materials. A company ASBERIT has tested CTA PAN-Ox material and were pleased to inform us that it can be used and they have a demand of 60 ton per year. The physical properties of this PAN-Ox are given in Table 17. As per the requirement of the ASBERIT company about 3.0 Kg of the processed PAN-Ox fiber were sent for testing at ASBERIT Co.

TABLE 17 - PHYSICAL PROPERTIES OF CTA OXIDIZED FIBER

Fiber nº	: CTA PAN OX-1
Tensile strength	: 0.175 GPa
Modulus	: 6.577 GPa
Density	: 1.498 g/cm^3
Denier	: 1.0888
Elongation	: 10%
Nº of filaments	: 18000
Tenacity	: 1.3 gm/denier
	1.29 gm/dtex
Breaking strength	: 1.57 gm
Filament dtex	: 1.2088
Heating temp.final	: 300°C, (first 230)
Residence time	: 120 minutes

4.0 - Developmental Activities in Surface Treatment

Surface treatment of carbon fibers is very important to form good composite with resin. The bonding of resin to the fiber depends mainly on the groups present on the surface of the carbon fibers. Poor bonding results, poor properties in final composites. The surface treatment to carbon fiber is done by different methods. For example, electrolytic oxidation, acid treatment alkali electrolysis, thermal oxidation, etc. In our group we have initiated surface treatment by electrolytic processo.

4.1 - Activities in 1982

At the time of my arrival, a few experiments were already conducted using nitric acid for the surface treatment of the carbon fibers. Due to non availability of uniformly processed carbon fiber, the developmental activities were postponed. However, attempts were made to acquire the materials and personnel for this development.

4.2 - Activities in 1983

With the formation of a group in surface treatment, the activities restarted. Anodic oxidation equipment was assembled by the CTA group in view of the information and assistance given by UNIDO experts, a few surface treatment experiments were conducted using CTA processed carbon fibers. A 20% surphuric acid was used as the electrolyte. About 125 meters of the fiber was surface treated. Unidirectional composites were made using epoxy resin DER 383 (Dow) and curing agent DEH 50 (Dow These composites were tested. In the subsequent experiments, the graphite electrode used earlier was replaced. Modifications were made using glassy carbon tube made out of polyperfural alcohol resin in CTA. Also subsequently several other modifications were made in the electrolytic cell. Also the experimental conditions such as currant density, electrolyte were changed and several of the experiments were conducted. Meanwhile, it is felt that we should get untreated carbon fibers from a commercial firm and surface treat them in order to standardize the surface treatment work. In view of our latest contacts with RK Textiles UK, sample of the untreated carbon fiber was obtained and surface treated with improved parameters. The Tables 18, 19, 20, 21 and 22 shows some of the results of surface treatment.

TABLE 18 - TYPES OF FIBERS USED FOR SURFACE TREATMENT

SAMPLE	TYPE OF FIBERS
1	PMR 428
2	PMR 429
3	PMR 432
4	SIGRAFIL
5	RK

TABLE 19 - DENSITIES OF SURFACE TREATED AND UNTREATED FIBERS

SAMPLE	NON SURFACE TREATED g/cm^3	SURFACE TREATED g/cm^3
1	1,75	1,78
2	1,75	1,76
3	1,75	1,79
4		1,78
5	1,74	1,74

TABLE 20 - PROPERTIES OF SINGLE FILAMENTS

SAMPLE	TREATMENT	TENSILE STRENGTH (GPa)	MODULUS E (GPa)
1	NT	2,4	190
	T	2,19	171
2	NT	2,4	190
	T	3,1	186
3	NT	2,7	190
	T	2,56	188
4	T	2,34	196
5	NT	3,25	222
	T	2,20	212

NT = Non-surface treated

T = Surface treated

TABLE 21 - PROPERTIES OF CARBON FIBERS/EPOXY RESIN COMPOSITES (VOLUME FRACTION: AROUND 60%)

SAMPLE	TREATMENT	FLEXURAL STRENGTH FS (MPa)	MODULUS E (GPa)
1	NT	1121	70
	T	988	80,5
2	T	973	79,5
3	NT	1614	117,5
	T	1100	81,2
4	T	1520	123,7

Table 22 - INTERLAMINAR SHEAR STRENGTH - ILSS

SAMPLE AND TREATMENT	ILSS (MPa)
2 T	45,3
3 T	42,0
4 T	54,1
5 NT	41,1
5 T (step 1)	49,8
5 T (step 2)	46,3
5 T (step 3)	40,6
5 T (step 4)	50,5

(Steps 1 to 4 = different current densities)

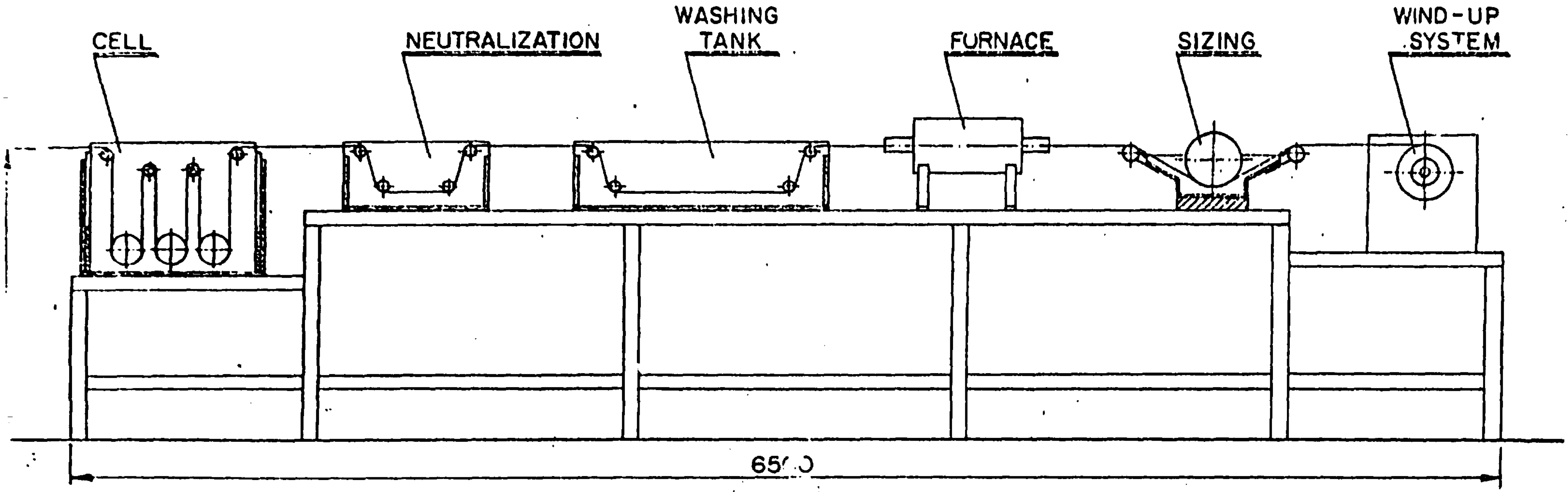
4.3 - Activities in 1984

i - Pilot Plant Surface Treatment Apparatus

Eventhough currantly batch scale surface treatment work is goin on in order to fix the surface treatment parameters, the final objective is to introduce a surface treatment equipment in line with the carbonization processing equipment. Such as equipment should be able to process 6 K tow at the rate of 1 to 30 m/h. After a detail analysis a cell was designed. Contacts were also made with local companies for the construction of this cell. This cell after completion will look like figure 10.

ii - Sizing

An equipment has been fabricated for the sizing of the surface treated carbon fibers. This will also go on line in the total carbonization equipment. A few batch scale experiments were already performed for sizing using 2% epoxy resin DER 757 (Dow) dissolved in acetone..



igure 10- LAY-OUT OF THE CARBON FIBRE PILOT PLANT SURFACE TREATMENT UNIT

5.0- CONFERENCE IN APPLICATIONS OF CARBON FIBERS

In order to expose the local scientists about the extensive applications of carbon fibers and to consolidate hitherto obtained results in PMR/CTA, an international conference on the applications of carbon fibers was planned to ~~be held in CTA during Dec 1983 under the sponsorship of CTA,~~ UNIDO and UNFSSTD. About nine experts in the field were invited to give conference lectures on applications of carbon fibers in their respective countries. The deliberations were held in CTA from 5th Dec - 7th Dec. Also participants from developing countries like Argentina, China, Egypt, Korea and Mexico have participated and benefited by the conference procedings. About 212 participants from Brazil attended the conference. On 8th and 9th Dec UNIDO, UNDP, UNFSSTD representatives together with the invited experts visited FISIBA to witness the developmental activities on UNIDO supplied pilot plant spinning assembly. The final conference report is enclosed as Annex III.

6.0- PROJECT ACTIVITIES - GENERAL

The general aspects of Project developmental activities were reported to UNDP in form half yearly progress reports and to UNIDO in form Project Evaluation Reports.

7.0- ANALYSIS

Detailed analysis with respect to the outputs activities and objectives were brought out in the latest UNIDO Project Evaluation Report, Annex IV. The comments of UNIDO Project Evaluation unit are worth to note on 1984 PER, ie Annex IV.

8.0 - ADDITIONAL OUTPUTS

The additional outputs achieved but not mentioned in the Project Document are worth noting.

i - For pilot line of carbon fiber processing, a new building became necessary and was built.

ii - New clean conditioned laboratories were built for chemical, analytical and testing purpose within the Carbon Fiber Project.
PAN-Ox activities with ASBERIT Co have started.

iii - Utilities for the pilot plant carbonization furnace were supplied by CTA.

iv - A group of experienced personnal in composites were included who would work in the 2nd phase on applications of carbon fibers, if approved.

v - An international conference on carbon fibers conducted with the sponsor ship of CTA/UNIDO/UNFSSTD.

vi - Additional testing and analytical apparatus not mentioned in the Project Document were developed, such as: denier meter, flash evaporator, fiber density measuring equipment, equipment to measure critical solution time, etc.

vii - 2nd phase document on applications of carbon fibers completed and sent to the Government for the submission to UNDP/UNIDO.

9.0 - INDUSTRIALIZATION OF CARBON FIBER TECHNOLOGY

Industrialization activities involvement of COPENE - Petroquímica do Nordeste S.A.

COPENE - Petroquímica do Nordeste S.A. is interested in scaling up activities. In view of this, visits were made from both sides ie CTA and COPENE. A document prepared by COPENE is under examination at CTA. COPENE is very much enthusiastic to scale up carbonization and in producing PAN-Ox.

10.0- ANALYSIS ON COUNTERPART SCIENTISTS SELF-RELIANCE AND INDEPENDENCE

In course of 2.1/2 years of my stay, I have seen the steady growth in the counterpart scientist's confidence and self-reliance. They have improved very much in solving the problems by themselves. Now I can see a good change from dependence to independence. At the beginning, everything was taken for granted and was done as told. Slowly this changed from how to why. The counterpart scientists are looking answers for questions. This gave them self-thinking capacity.

The initial dependence on experts has declined in course of time as one may notice from the figure 11. The counterpart scientists who were trained at CTA are helping the people in FISIBA in scale-up activities. The initial and first paper (Annex 1) consisting CTA results were presented by CTA counterpart scientist in 1983 with a maximum help and backing of international experts. In 1984, the counterpart scientists gained confidence and presented additional results (Annex II) in France, with minimum assistance and backing. This shows the confidence of the counterpart scientist.

Based on the knowledge gained due to the visits of UNIDO experts, the counterpart scientists are now thinking new areas of research within this Carbon Fiber Group.

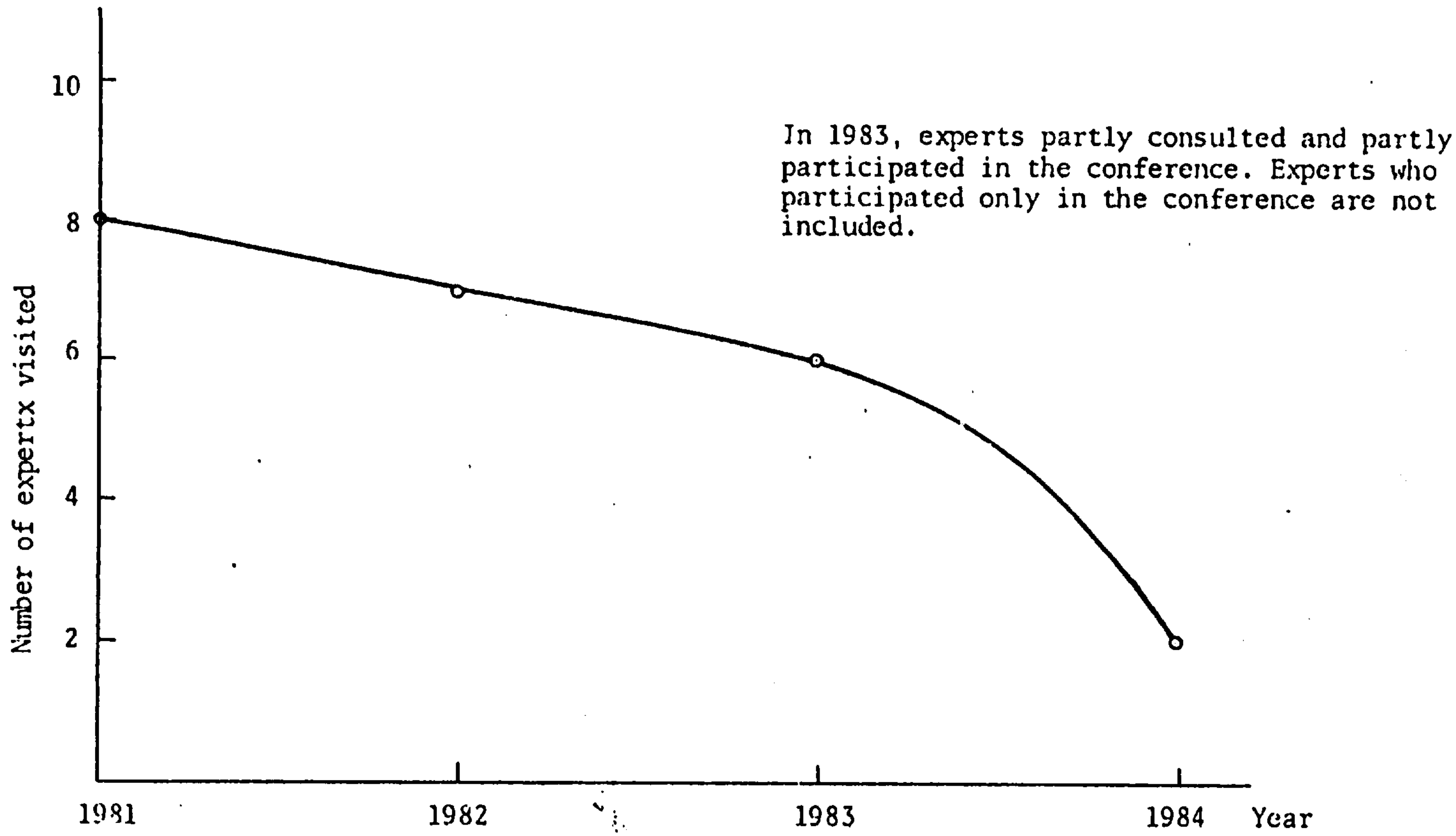

In 1983, experts partly consulted and partly participated in the conference. Experts who participated only in the conference are not included.
Number of expertx visited
10
8
6
4
2
1981
1982
1983
1984
Year

Figure 11.

11.0- RECOMMENDATIONS

i - In R & D Activities

- The Precursor Group should synthesis more polymers as mentioned in Chapter 2.3 and should characterize the same.
- The Precursor Group should spin acrylic polymers and develop new spinning procedures and develop new parameters as mentioned in Chapter 2.4.
- The Precursor Group should advise these to FISIBA.
- The Heat Treatment Group should work with pilot plant C-fiber plant continuously for week at least without stopping.
- The Heat Treatment Group should process the commercial acrylic fibers for PAN-Ox applications, and also explore industries to use carbon fiber on commercial level application such as activated filters, etc.
- The Surface Treatment Group should introduce the surface treatment equipment in the line as soon as possible.
- The composites group should be given materials and analyse the processed carbon fibers in form of composites.
- Whenever necessary, scientific workshops may be conducted to appraise the latest development in this technology.

ii - General Nature

- The 2nd phase on applications of carbon fibers should be started in continuation of the present project in order to consolidate the achievements obtained in the present project.
- All attempts should be made not to loose the so far UNIDO trained counterpart scientists in the development of this technology.
- The terms for cooperation with COPENE to industrialize the carbon fiber technology developed in this Project should be finalized as early as possible. Necessary assistance should be given to COPENE in their venture to industrialize carbon fibers.

ooo 0 ooo

INFLUENCE OF PROCESSING VARIABLES ON CARBON FIBRE PROPERTIES IN A CONTINUOUS PROCESSING SYSTEM

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INTRODUCTION

Young's modulus and tensile strength of carbon fibres depend strongly on the preferred orientation of carbon layers in the fibres (ref. 1, 2, 3). The preferred orientation of these layers can be increased by stretching the fibres during all stages of C-fibre production. In case of PAN-based C-fibres the prestretching of the initial PAN-fibres is most effective in order to increase modulus (ref. 4, 5) and tensile strength (ref. 5, 6). Tensile strength, however, is influenced by defects in the fibres, which can be created in all steps of the process. Especially the oxidation of PAN is very sensitive with respect to the creation of defects, so that the various parameters of this step must be optimized in order to obtain high quality carbon fibres (ref. 7, 8, 9).

This paper is concerned with the influence of the degree of prestretching and oxidation process parameters on intermediate products and on the properties of resulting C-fibres from PAN.

EXPERIMENTAL

A commercially availabe special grade PAN-fibre (S.A.F.1/6K/1.22 from COURTAULDS, ref. 10) was used for the studies. The processing into carbon fibres was done in four subsequent continuous steps:

A- Prestretching from 0 to 15% in hot air at 190°C
B- Oxidation at constant length in hot air at 230°C
C- Oxidation at constant length in hot air at 265°C
D- Carbonization up to 1100°C in argon, allowing a shrinkage of about 7%.

For the steps A, B and C a tube furnace of 3 m length was used, through which the fibres have been drawn by roller system. In the oxidation steps B and C the amount of forced air and the residence time have been varied. For carbonization (step D) the fibres have been drawn through a tube furnace purged with argon without variations of residence time and final temperature.

The properties of the fibre as density, young's modulus, tensile strength and the mass changes were determined after each processing step. For density measurements a float sink method was used (ref. 5). The mechanical properties of the fibres were measured on an Instron at a gauge length of 25 mm and a cross head speed of 2,5 mm/min. in case of C-fibres and 10 cm/min. for the other fibres.

RESULTS

The increase in fibre density gives information on the progress of oxidation. As can be seen in Figure 1, during the prestretching at 190°C (step A) nearly no oxidation occurs. During step B (oxidation at 230°C) the density increased by 0,15 g/cm^3. Applied forced air flow reduces the density increase. The forced air flow causes an opposite effect on the further density increase during step C. Nearly no influence of air flow on the total density increase after steps A to C can be detected.

The young's modulus of the precursor fibre decreased continuously during the steps A to C (Table 1). Forced air flow diminishes this decrease. Increased preferred orientation achieved by prestretching in step A, causes higher absolute values for the modulus, while the highest applied prestretching (15%) reduces the modulus again slightly.

The influence of prestretching on the modulus of the resulting carbon fibres is shown in Figure 2. As expected, the modulus increases with increased degree of prestretching. Increased air flow during oxidation steps B and C diminishes the modulus of C-fibres for lower degree of prestretching.

SUMMARY

An influence of the investigated process variables, prestretching and forced air flow the oxidation steps can be found at the intermediate products and on the finally resulting carbon fibres. Changes in the air flow mainly influence the intermediate products after step B and C as found by the density measurements. No or low air flow rate accelerates the strongly exothermic cyclization in step B autocatalytically by the unsufficient heat transfer from the fibre to the air. The oxygen uptake during oxidation of PAN requires at least partly cyclized fibres (ref.5, 11), so that it can be assumed that it occurs mainly during step C. High air flow rates facilitate this difusion controlled rates.

The increasing modulus of fibres after the oxidation step B and C support these explanations.

The degree of prestretching influences mainly the modulus of the resulting carbon fibres. Increasing preferred orientation achieved by prestretching of the PAN-fibre increases the modulus.

ACKNOWLEDGEMENT

The authors are highly thankful to Prof. E.Fitzer, University of Karlsruhe, W. Germany for his constant encouragement, inspiration, sustained interest and for his valuable comments in the work, to T. Cal Kessel, Chief of the Division for his interest, support and facilities and to United Nation Industrial Development Organization (Vienna) for sponsoring the project.

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** UNIDO expert, formaly was at Institute Fur Chemische Technik, University of Karlsruhe, West Germany.

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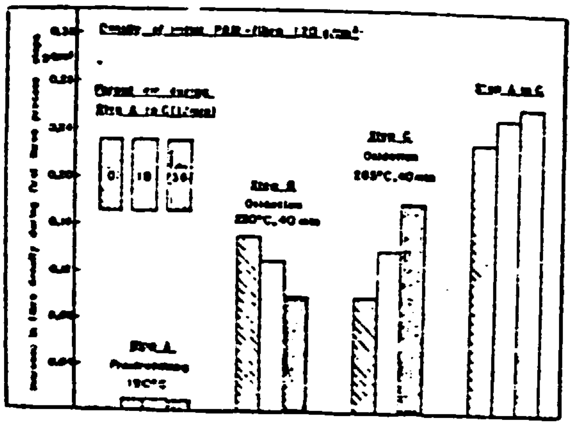

Figure 1. Increase of fibre density after process steps A, B and C.

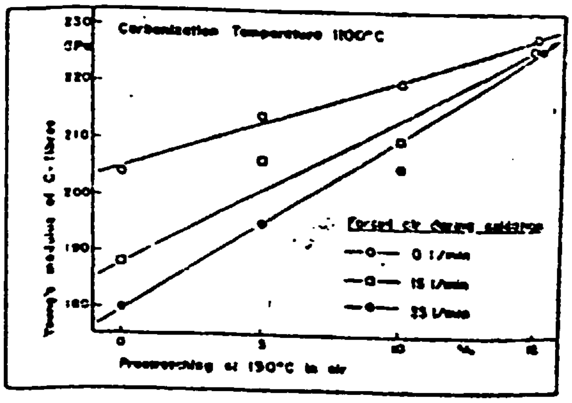

Figure 2. Influence of prestretching on the young's modulus of resulting carbon fibres.

Table 1. Young's modulus of fibres after process step A, B and C (Initial modulus of PAN precursor fibres was 10.8 GPa)

FORCED AIR FLOW RATE L/MIN.)		0				15				35			
DEGREE OF PRESTRETCHING (%)		0	5	10	15	0	5	10	15	0	5	10	15
YOUNG'S MODULUS OF FIBRES (GPa)	STEP A	10.0	11.1	11.7	11.4	8.5	10.0	9.8	8.9	10.1	10.6	11.6	10.3
	STEP B	7.1	7.6	7.2	6.8	7.5	8.5	7.7	6.9	7.6	9.5	9.3	[illegible]
	STEP C	6.4	6.7	6.8	6.4	6.4	7.3	7.2	6.0	7.6	7.6	7.5	6.9

CARBON FIBRE CHARACTERISTICS OBTAINED FROM STABILIZED PAN IN NO_2 ATMOSPHERE

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SYNOPSIS

It is shown that NO_2 decreases the time required to stabilized PAN fibres suitable for carbon fibre production. There is also a modest improvement in strength. NO_2 in excess, however, is found to be deletereous. It merely modifies the chemical kinetics of the stabilization process and is not incorporated in the structure of the material.

INTRODUCTION

An important step in the production of good quality carbon fibres is the stabilization of the PAN precursors at 265°C in air. This process is rather slow for continuous fibre manufacture and requires acceleration in commercial applications. Marinkovic (1) and Bahl (2) have suggested the use of SO_2 at this important stage. It is reported that SO_2 accelerates the stabilization process and improves the strength of the carbon product to some extent. It is also claimed that the SO_2 is incorporated in the surface of the fibres. In many ways NO_2 behaves chemically in a similar way to SO_2 and so we decided to experiment with this oxidizing agent to improve the rate of continuous fibre production in our carbon fibre plant.

EXPERIMENTAL DETAILS

In this series of experiments we used polyacrilonitrile 1/6K/1.22 produced by Courtaulds. There were three stages in the process:

Ia - Either stabilization in air for 40 min. at constant length at 230°C, or
Ib - Stabilization in NO_2/air mixtures for a range of residence times at constant length also at 230°C,
II - Stabilization in NO_2/air mixtures for a range of residence times at constant length at 265°C,
III- Carbonization in argon, allowing shrinkage to 1000°C.

For stages I and II we used furnace, lm long with a device for maintaining the length constant. For stage Ia the residence time was 40 minutes (ref.3). In stage Ib we used NO_2 in concentrations of 5 to 30% in air and residence times of 10 to 60 minutes. In stage II we used NO_2 in concentrations of 10 to 40% in air and residence times of 10 to 60 minutes. In stage III, the residence time was 60 minutes with a shrinkage of between 7 and 14%. After each stage of stabilization, specimens were analysed by DSC (Dupont) and IR (Perkin Elmer) to determine the degree of stabilization. Density measurements were also made to this end. The tensile strength and Young's Modulus were determined only after step 2 using an Instron with cross-head speed of 10mm per minute. After stage 3, the density, strength and Modulus of elasticity were measured to determine the quality of the carbon fibre produced. The cross-head speed of the Instron, in this case, was 2.5mm per minute.

RESULTS

Some of results are summarized in tables I-II-III and figure 1.

DISCUSSION

It is interesting to compare the results in tables I and II with those of table III (weight loss). The highest weight loss during carbonization occured after the use of high concentrations of NO_2 (40%) during stabilization, bringing with it a marked reduction in mechanical properties (probably because of structural damage). It is interesting to determine why high concentrations of NO_2 produce such drastic deteriorations in mechanical properties of fibres whereas lower concentration of NO_2 produce modest improvements in strength (table I). Moreover, carbon fibres with poorer mechanical properties are produced when stabilization takes place in shorter times using an atmosphere of NO_2 in air in both stages of stabilization. In the IR analysis, it is noted (figure 1) that there is a tendency to reduce the removal of the nitrile groups in the presence of NO_2. This decrease is proportional to the concentration of NO_2. In stage II, however, it may be seen that all the analyses made at every stage did not show the presence of any group containing nitrogen, showing that no nitrogen groups are incorporated in the molecular structure of the treated fibres.

CONCLUSIONS

Treatment with NO_2 like SO_2, produces a modest improvement in the strength of carbon fibre and accelerates the stabilization stage in continuous fibre production. NO_2 alters the chemical kinetics in the stabilization of PAN fibre, but in contrast with SO_2, is not incorporated in the PAN structure.

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TABLE I- Tensile strength of carbon fibres after carbonization (1000°C)

% NO_2	Oxidation Time (min.)					
	10	20	30	40	50	60
0	1,54	1,58	1,93	2,16	2,04	1,72
10	1,42	1,92	1,91	2,06	1,86	1,59
20	1,70	1,93	2,23	2,18	1,79	1,86
30	1,97	1,70	1,70	1,68	1,60	1,48
40	1,76	1,38	1,30	1,15	1,16	1,07

TABLE II- Young's Modulus of carbon fibres after carbonization (1000°C)

% NO_2	Oxidation Time (min.)					
	10	20	30	40	50	60
0	147	169	163	142	124	121
10	148	160	157	148	151	127
20	153	157	169	160	158	155
30	160	149	153	148	144	141
40	158	137	142	111	112	107

TABLE III- Weight loss % after carbonization (1000°C)

% NO_2	Oxidation Time (min.)					
	10	20	30	40	50	60
0	37,9	37,9	41,6	40,8	38,9	39,4
10	36,5	37,9	37,9	37,9	38,1	39,1
20	39,4	39,6	39,7	40,3	40,3	41,7
30	38,0	37,9	37,9	39,6	41,7	41,9
40	42,0	42,4	43,9	44,3	45,2	48,4

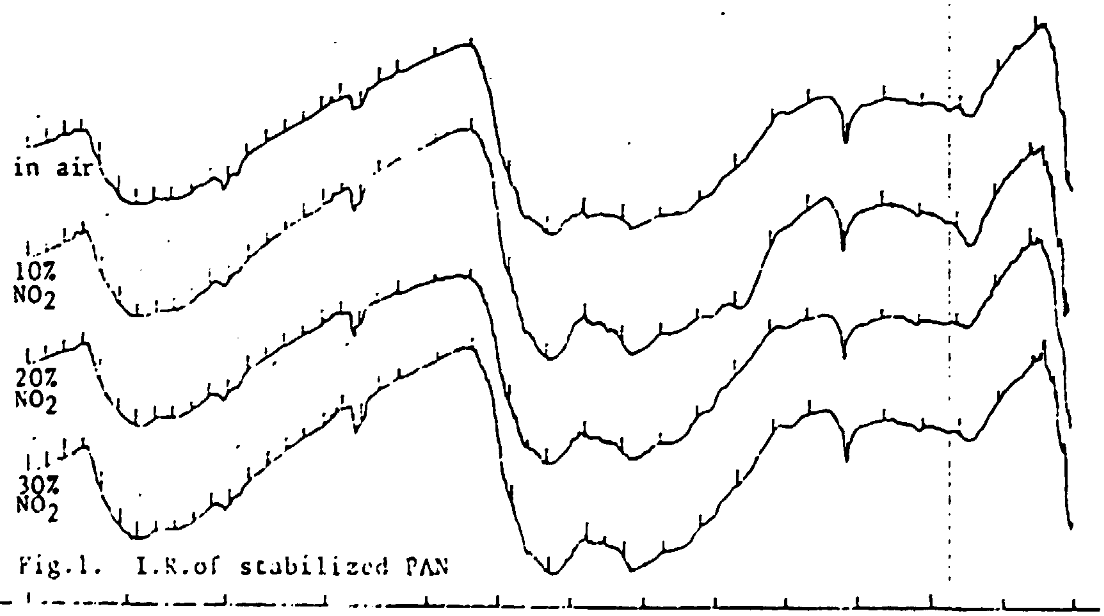

Fig.1. I.R.of stabilized PAN

FINAL REPORT OF THE "INTERNATIONAL CONFERENCE ON CARBON FIBER APPLICATIONS"

The Government of Brazil, through the Centro Técnico Aeroespacial (CTA), organized and hosted an International Conference on Carbon Fibre Applications, from 5-9 December 1983, at São José dos Campos (SP) and Camaçari (BA). The Conference was co-sponsored by the United Nations Financing System for Science and Technology for Development (UNFSSTD) and the United Nations Industrial Development Organization (UNIDO).

The purpose of the Conference was, on the one hand, to present the results of a successful project on "Optimization and Development of Carbon Fibre Technology", which is being implemented at CTA with the financial co-operation of UNFSSTD and the technical assistance of UNIDO, and share such results with representatives of invited developing countries which have reached a similar technological and industrial level so that they are interested in the production and/or use of carbon fibres and composites.

On the other hand, the Conference served the purpose of demonstrating the great variety of applications of carbon fibres and composites in aerospace and transportation as well as in marine, recreational and biomedical uses. This was achieved through the co-operation of a group of high level experts from some developed countries.

Finally, the Conference focused, in more general terms, on the identification of methods and mechanisms on which an efficient technical co-operation among interested developing countries would be based with a view to sharing the experience which each of these countries has with regard to production and/or applications of carbon fibres and composites and, thus, strenghten the scientific and technological capacity of these countries.

China has been working, since aproximately 1960, in the development of carbon fibre technology and its industrial applications and the development and production of resins for composites with the fibre.

India, with the help of UNIDO, developed competence for the production of appropriate precursors for carbon fibre, the production of the fibre itself and its use in many industrial applications. At present, India is making a strong effort to acquire capacity to internally produce resins for more sophisticated applications of the fibre.

Brazil, with the financial support of the UNFSSTD and having UNIDO as the executing agency, has developed capacity for the production of the PAN precursor, to produce carbon fibre and to produce composites for many industrial applications with the fibre. It is expected that, by the end of 1984, Brazil completes the development of the knowledge for the production of carbon fibre up to the industrial scale.

Korea

Argentina, Egypt and Mexico have been using carbon fibre composites in industrial applications since many years.

Through all those activities, a substantial capacity, in the chain that goes from the production of the precursor for the carbon fibre up to its application, has been developed in those developing countries; a substantial amount of information has been acquired or generated. The levels of capacitations acquired by each country is different from country to country in the different parts of the chain, though they can be complementary.

In spite of all the efforts being made, there are big tasks to be performed by those countries to complete their capacities in this field and, from there on, to cope with new developments in the field. All this can be quite costly and will need sustained international assistance.

In view of all those facts, it is clear that there exists the evident possibility for the developing countries attending this meeting to cooperate.

In this context, UNFSSTD and UNIDO understand their role as that of providing an interactive mechanism to help to catalyze co-operative activities of developing and developed countries - of governments, universities, research laboratories, public and private enterprise and financing institutions - with a view to finding appropriate solutions for the problems of acquisition, assimilation and application of carbon fibre and composite technology for the benefit of the economic and social development of those countries.

The efforts of the countries, and the external support by the United Nations Systems, should be able to draw on co-ordinated services from every interested country that can provide them, to any interested country that needs them, and they must be able to do this on a reliable and sustained basis.

In this spirit, participants from Argentina, Brazil, China, Egypt, (India), Korea and Mexico have discussed possible subjects and mechanisms of co-operation in the field of production and application of carbon fibres and composites and have come to the following conclusions:

1. With a view to gaining the full support of the competent Government authorities of the countries represented at the Conference, the participants agree to establish the necessary contacts to such authorities, inform these fully on the results of the Conference and recommended follow-up activities and convey to them the importance of co-operation in this field.

2. In order to assure that each participating country be fully informed on relevant activities related to the production and application of carbon fibres and composites undertaken in the other participating countries, the participants identify the need for an information network covering such activities which, in the first stage of co-operation, could function on an informal basis. As a first step, such network would gather and disseminate the information actually available in each participating country on past or ongoing activities and experiences.

3. With a view to providing training facilities for scientists and technicians from developing countries, the participants agree that existing R & D and production centres, such as CTA and FISIBA in Brazil, should receive trainees from interested countries, subject to specific agreements. In order to maximize training results, each regional centre which receives the visit of high level experts from developed countries should consider to invite participants from other interested developing countries to the training activities.

4. Taking into consideration the absence of norms and standards in the field of carbon fibres and composites, the participants agree to the need to develop common standards for their countries so as to enable them to exchange raw materials, intermediate and end-products of common specifications and of controlled quality. For the same purpose, common rules for certification should be elaborated.

5. With regard to the above identified needs and others, which the internal consultations of each participating country may consider; the participants of this Conference see an important role for the United Nations System, especially UNFSSTD and UNIDO, to provide the interactive mechanisms for the identification and mobilization of the needed technical and financial resources.

ANNEX IV

Project Evaluation Report

Part I — Basic Data

Performance (II) Terminal (III)	Project Title (from project document): OPTIMIZATION & DEVELOPMENT OF CARBON FIBER TECHNOLOGY
Backstopping Branch/Section: / CHEM	Total project budget ($US): (latest signed revision line 99) 1,354,954 Cost sharing contribution (if any):

project approved: [illegible]/05/83	Period covered by this evaluation report: From: Dec 2, 83 To: 29 June 1984	Participants (check one or more): ☒ CTA ☒ National counterpart ☐ Other international project staff ☐ Other SIDFA
operations commenced: [illegible] 82		
planned/actual completion date: Dec 1984	Date of last evaluation/PER Nov 29, 1983	

ACTIONS REQUIRED

Agency responsible	Priority Action/Decision/Follow-up required or recommended as a result of the evaluation	Target deadlines
[illegible]	To provide utilities in the new building for carbon fiber pilot plant.	Aug 1984
	Provision to work continuously with the pilot plant for a week without stopping.	Aug 1984

Prepared by:

Chief Technical Adviser T. NAGABHUSHANAM (Name)

[illegible] ROBERTO KESSEL (Name and title)

Date [illegible] June 1984

Endorsed by (see page 5 for comments):

1. SIDFA [signature] (Name)
Date 10 – VIII [illegible]

2. Backstopping Officer [illegible] Kessel (Name)
Date 12 September 1984

Reviewed by:

3. Branch Head A. [illegible] (Name)
Date 12 September 1984

UNIDO

INTERNAL EVALUATION SYSTEM

Project Evaluation Report

Part II — PERFORMANCE ASSESSMENT
(Self-evaluation)

Required only for [illegible] large scale projects on an annual basis until completion. No evaluation of progress is required [illegible] projects [illegible] Part II may be [illegible] at management's [illegible] for example, extended duration of project [illegible] for [illegible] purposes. [illegible] UNIDO Secretariat and field staff and for guidance on preparation of [illegible]

PERFORMANCE ANALYSIS

A. Causal Factors for Analysis

Please complete attached checklist of causal factors before proceeding further.

B. Progress in Producing Individual Outputs/Results

How many outputs are listed in the project document? 4

Output No.1

1. Repeat as appears in latest Prodoc/PDS or as modified by tripartite decision (specify if not from project document):

   A research and development team fully trained and with the experience to keep adapting the technology used in other countries to produce carbonfibers or related materials, or to further improve the technology already in use in Brazil, or to conduct R and D work related to other similar materials from the laboratory scale to the industrial scale.

2. Progress (check one):

   ☐ none ☐ slow/poor ☐ adequate ☒ very good ☐ excellent ☐ cannot determine

   Explain:

   See enclosure 1

3. Factors impeding progress (check one):

   ☐ slow delivery of inputs ☐ poor quality of inputs ☐ inadequate work programme
   ☐ change in critical assumptions (external factors) ☐ other unanticipated events ☒ no problems/on schedule

   Explain:

4. Possibility of producing intended results/outputs in quantity, quality and time originally expected (check one):

   ☐ impossible ☐ improbable ☐ likely ☐ highly likely ☒ certain ☐ cannot determine

   Explain:

   See enclosure 2.

ENCLOSURE 1

Counterpart group is more seriously involved in the developmental activities. Completed bench scale standardization in heat treatment and spinning. Now in the process of scaling up. New oxidising systems studied. New spinning experiments based on preliminary carbonization studies of CTA precursor have already commenced. Methods improved in surface treatment experiments with sizing systems commenced. The training of one of the counterpart scientists in USA through UNID helped him to fabricate prepreg laminate equipment and analysis. T.Cel Kessel counterpart group leader's study tour with two of his collegues helped him in advising his group at CTA what he gained in the tour.

ENCLOSURE 2

Bench scale carbonization and spinning experiments almost completed. The team has been trained by the competent experts. The overall world activities in this field were exposed to the team by conducting [illegible]ational symposium in carbon fibers and by inviting experts competent in the field. Pilot plant equipment arrived. Work already started to scale up. The counterpart scientists went abroad on study tours and fellowship to get themselves acquinted with latest developments in the field. Two of the counterparts are attending a conference in France and presenting a paper in carbon conference. Another counterpart scientist is to attend a UNIDO's implant group training in Austria in Oct/Nov 84. National Coordinator and two of his collegues recently visited Europe on study tour which helped them in adapting to the local needs. Thus the team has formed and exposed to the activities elsewhere in the world and adapting to the local needs.

Output No.* 2

1. Repeat as appears in latest Prodoc/PDS or as modified by tripartite decision (specify if not from project document):
   See enclosure 3.

2. Progress (check one):
   ☐ none ☐ slow/poor ☐ adequate ☐ very good ☐ excellent ☐ cannot determine
   Explain: See enclosure 4.

3. Factors impeding progress (check one):
   ☐ slow delivery of inputs ☐ poor quality of inputs ☐ inadequate work programme
   ☐ change in critical assumptions (external factors) ☐ other unanticipated events ☐ no problems/on schedule
   Explain:

4. Possibility of producing intended results/outputs in quantity, quality and time originally expected (check one):
   ☐ impossible ☐ improbable ☐ likely ☐ highly likely ☐ certain ☐ cannot determine
   Explain: See enclosure 5.

Output No.* 3

1. Repeat as appears in latest Prodoc/PDS or as modified by tripartite decision (specify if not from project document):
   See enclosure 6.

2. Progress (check one):
   ☐ none ☐ slow/poor ☐ adequate ☒ very good ☐ excellent ☐ cannot determine
   Explain: See enclosure 7.

3. Factors impeding progress (check one):
   ☐ slow delivery of inputs ☐ poor quality of inputs ☐ inadequate work programme
   ☐ change in critical assumptions (external factors) ☐ other unanticipated events ☐ no problems/on schedule
   Explain:

4. Possibility of producing intended results/outputs in quantity, quality and time originally expected (check one):
   ☐ impossible ☐ improbable ☐ likely ☐ highly likely ☐ certain ☐ cannot determine
   Explain: See enclosure 8.

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* [illegible]

ENCLOSURE 3

Technology to produce carbon fibers of the appropriate quality and the corresponding precursor, using national raw materials. All this technology transferred to the industrial system.

ENCLOSURE 4

Carbon fibers with comparable properties with those of commercial fibers were produced. Acrylic fibers of commercially comparable properties were also produced. The scaling up to pilot plant already commenced. All the utilities to pilot plant carbonization furnace are being completed. The acrylic fibers produced from national raw material are being standardized for carbon fiber processing. The team is capable of analysing new raw materials for carbon fiber processing and fiber spinning.

ENCLOSURE 5

Good quality carbon fibers and precursors fibers already developed in the bench level. Scale-up activities already commenced. The industry already involved. COPENE - Petroquímica do Nordeste S.A., the parent organization of FISIBA - Fibras Sintéticas da Bahia S.A. is very much interested in these developmental activities.

ENCLOSURE 6

Knowledge and techniques to characterize and test the raw materials as well as the final products for the production of the precursor and carbon fiber.

ENCLOSURE 7

The team has characterised the raw materials available precursor fibers and has been conducting experiments to analyse the intermediate and final products. Also characterised the commercially available final products.

ENCLOSURE 8

The team is now capable of performing this activity confidently. Acquired required knowledge to characterise the raw materials and final products.

Output No.* 4

1. Repeat as appears in latest Prodoc/PDS or as modified by tripartite decision (specify if not from project document).
See enclosure 9.

2. Progress (check one):
☐ none ☐ slow/poor ☐ adequate ☒ very good ☐ excellent ☐ cannot determine
Explain: See enclosure 10.

3. Factors impeding progress (check one):
☐ slow delivery of inputs ☐ poor quality of inputs ☐ inadequate work programme
☐ change in critical assumptions (external factors) ☐ other unanticipated events ☒ no problems/on schedule
Explain:

4. Possibility of producing intended results/outputs in quantity, quality and time originally expected (check one):
☐ impossible ☐ improbable ☐ likely ☐ highly likely ☒ certain ☐ cannot determine
Explain: See enclosure 11.

Output No.* ____

1. Repeat as appears in latest Prodoc/PDS or as modified by tripartite decision (specify if not from project document):

2. Progress (check one):
☐ none ☐ slow/poor ☐ adequate ☐ very good ☐ excellent ☐ cannot determine
Explain:

3. Factors impeding progress (check one):
☐ slow delivery of inputs ☐ poor quality of inputs ☐ inadequate work programme
☐ change in critical assumptions (external factors) ☐ other unanticipated events ☐ no problems/on schedule
Explain:

4. Possibility of producing intended results/outputs in quantity, quality and time originally expected (check one):
☐ impossible ☐ improbable ☐ likely ☐ highly likely ☐ certain ☐ cannot determine
Explain:

* For any additional outputs as given in the project document add supplemental pages as necessary using same form

ENCLOSURE 9

Physical facilities completed in such a way that the trained staff can properly perform their tasks. The additions in this second phase will include:

- pilot equipment for spinning the polyacrilonitrile precursor;
- modified pilot line (including surface treatment) for the production of carbon fibers;
- auxiliary equipment for electron microscopy X-ray diffraction, differential thermal analysis, electronic measurements, photography;
- other minor equipment.

ENCLOSURE 10

Achieved almost as far as required for the project.
Pilot plant for spinning polyacrylonitrile precursor acquired,work already commenced on this equipment at industrial site at FISIBA, Salvador.
Equipment for pilot line acquired. The utilities are almost completed.
Preliminary activities are being commenced.
Most of the auxiliary equipment as for required to the project obtained.
Most of the minor equipment required has been purchased or fabricated by CTA.

ENCLOSURE 11

Most of the factors in this output achieved. The remaining are sure of achieving.

C. Critical Assumptions

1. Briefly describe those assumptions, explicit or implicit which, while outside of project management control, are critical to production of one or more of the outputs listed above (e.g. projected industry demand or need).

Conference on carbon fiber applications to appraise the local industrial involvement of the local industries to produce carbon fibers involvment of local industries which could use these fibers.

2. For each assumption, describe what has or has not taken place, or seems most likely to take place, and the actual or possible effect of such change on producing the outputs/results originally intended (e.g. if industry demand has not developed as projected, what is the probable cause), including what remedial actions, if any, can or should be taken by any of the tripartite parties:

A conference was conducted on application of carbon fibers. About 200 participants from industries, institutes, government organization appreciated the application of these fibers.
A major pretrochemical industry COPENE - Petroquímica do Nordeste S.A., the parent organization of FISIBA has shown interest in the industrialization of these fibers and visited the project site to have the first hand information of this technology. The neighbouring civil commuter plains manufacturer EMBRAER expressed its plans of using carbon fibers. All these are positive effects for the project.

ASSESSMENT

D. Project (hypothesis) Approach and Design

1. Repeat project (immediate) objective as it appears in latest Prodoc/TDS, or as subsequently modified by tripartite decision:

See enclosure 12.

2. Given project experience to date, including the original assumptions made and any changes which have occurred in the project environment, is the project hypothesis still valid, i.e. if the outputs are produced as planned then is it reasonable to assume that the project objective(s) will be successfully achieved?

☒ yes ☐ no, explain ☐ cannot determine, explain

Most of the outputs are achieved.
A strong enthusiastic group has formed.
Most of the technology on bench scale completed, industry already involved.
Knowledge on techniques is acquired by the group.
Most of the physical facilities were provided.
The remains are sure of achieving.

ENCLOSURE 12

The immediate objectives are:

i- to complete the formation of the scientific and technical capability to be able to industrially produce carbon fiber precursors, carbon fibers and its composite structural materials and to further develop the technologies involved in that process, from the laboratory scale up to the industrial scale. This means training the appropriate personnel and forming the corresponding teams, not only for the production process for the carbon fiber, but also to determine the chemical, physical and mechanical characteristics of the raw materials, intermediate and final products;

ii- to optimize the continuous process for the production of carbon fiber which has already been developed up to the bench scale by the CTA; to further develop it up to the pilot plant scale until sufficient knowledge and experience has been developed, so as to transfer it to the production system;

iii- to generate the information necessary to produce the precursor fiber and to transfer it to the production system;

iv- to complete the minimum necessary equipment and physical infrastructure in such a way that the teams formed through the project can appropriately perform their tasks.

3. Given the status of producing the individual intended outputs/results as indicated above, what is your judgement of the probability of successfully producing the overall final results reasonably on schedule, within the resources to be supplied, and in the quantity, type and quality required?

☐ impossible ☐ improbable ☐ likely ☐ highly likely ☒ certain ☐ cannot determine

4. Is a project redesign necessary? ☐ yes ☒ no

5. If yes, at what level?

☐ development level ☐ project approach/all levels ☐ development hypothesis

☐ outputs ☐ project (immediate) object ☐ work programme

E. Overall Conclusions and Recommendations

1. On the basis of your ratings above, provide a concise narrative evaluation statement of progress to date, including any relevant and significant information not included above:*

The group has become strong both in number and kind. Thinking capacity of the group has improved. The counterpart scientists are capable of analysing the problems. The self confidence in the counterpart scientists is growing. This is a overall positive approach in achieving the goals and objectives of the project. The leadership of T.Cel Kessel was given morale to the group. The enthusiasm and encourage given to the group personell is yet another positive factor.

Also see enclosure 13.

2. Describe fully the actions or decisions recommended by project management to improve the efficiency and effectiveness of project operations, including responsible units and suggested target dates.* Summarize under "Actions Required" on Part I — PER Face Sheet:

The "Actons required" in the previous PER were accomplished. Centro Tecnico Aeroespacial inducted the more qualified personnel into the group and completed the most of the facilities. Now in the process of providing utilities like inert gas, refrigeration, compressed air, etc. UNIDO has purchased all of the requirements requested by CTA.
It is now recomended that Centro Tecnico Aeroespacial should complete providing the utilities as early as possible to use pilot plant carbonization furnace. Necessary arrangements must be made to run the carbonization furnace continuously, at least for week without interruption.
However, it is recommended to follow up this project activities with a 2nd follow up phase to consolidate and directly apply to industrial application.

Continued in Enclosure 14.

3. When was the last tripartite review or tripartite in-depth evaluation held? Tripartite review. Dec.2.1983
(date and type)

4. When is the next tripartite review or in-depth evaluation scheduled? November 1984. Tripartite review
(date and type)

5. Given the above conclusions, is an in-depth evaluation necessary in the near future (regardless of whether or not or when, one is included in the Prodoc or PDS)? ☐ yes ☒ no

*Attach additional pages as necessary

ENCLOSURE 13

In order to consolidate the activities and to expose others CTA conducted an international conference on application of carbon fiber during first week of December 1983, invited participants from other developing countries like, Argentina, China, Egypt, Korea and Mexico, and experts in the field from developed countries. All participants including those 200 participants from Brazil, appreciated the project activities at the project site.

The precursor group of the project has standerdised the spinning parameters, with respect to major spinning variables under DMF spinning conditions. Fibers comparable with most of the commercial fibers were produced from the polymer obtained from FISIBA. Qualitative experiments to scale up the spinning experiments have already been started at a fiber industry FISIBA in Salvador. A few fiber bobins spun from the pilot plant spinning assembly have been tested at CTA.

A heat treatment group has almost completed the newly designed experiments in carbonization as per the instructions of expert I.L.Kalnin. Also in order to explore other oxidising atmosphere in the oxidation stage, the group has completed oxidation studies using nitrogen dioxide. Currently systematic studies are being conducted to evaluate oxidation and carbonization properties for CTA spun fiber from FISIBA polymer. Various oxidazation temperatures, resident times were used. Parameters such as elongation, density, mechanical properties were determined. Also on the best oxidised fibers, carbonization studies already commenced. The shrinkage, wight loss, density, mechanical properties have been determined on same of these fibers. Spinning experiments are being modified based on these results. Active work is going on to assemble the pilot plant carbonization furnace. The required utilities are being provided.

Surface treatment experiments were conducted with a modified set-up and using untreated RK Company supplied carbon fibers. These studies would give an idea to standardize the surface treatment on CTA made carbon fibers. The treated fibers were used to unidirectional laminates using epoxy resins. The flexural, interlaminar shear strength, etc. were determined. Also sizing was introduced after the surface treatment as it is required to keep the fibers together.

Counterpart scientist , Álvaro Luiz de Sousa Gomes Filho was trained in USA on composites and analysis. As a result of this training, the fellow could develop vacum bag moulding for preparing prepreg laminates and develop analytical techniques.

T.Cel Kessel the counterpart Project Leader, went on study tour along with two of his collegues for discussions with persons in KK Company who are experienced in carbon fiber technology and who well oxidation ovens and carbonization furnace. T.Cel Kessel could have the first hand information on the possible purchase aspects of these equipment. T.Cel Kessel visited UNIDO for discussions with regards to the II phase project. This tour helped him in understanding and appreciating the potential of carbon fibers.

Some of experts who attended the Conference also consulted the group.

ENCLOSURE 14

Output nº 1 has already achieved. However, the team should be in constant contact with developmental activities elsewhere in the world. Further improvement in English language will help the counterpart scientist in interacting with people outside Brazil and could help in using the literature.

Output nº 2. Most of the output 2 achieved. However, the counterpart should new work on UNIDO purchased pilot plant carbonization furnace and should work continuously a 24 hrs basis, and should produce PAN-ox and carbon fibers.

Output 3. This has already been achieved.

Output 4. As far as required have been completed. However, since the pilot plant carbonization furnace arrived recently all the utilities should be completed as soon as possible to work on this equipment.

[illegible]

[illegible] SIDFA and [illegible] backstopping officer [illegible] findings, conclusions and recommendations of project management [illegible] differences and/or [illegible]

SIDFA Backstopping officer

☐ ☐ completely endorse PER

☑ ☐ endorse with following comments and/or reservations:

☐ ☐ do not agree with assessment or recommendations as explained:

1. SIDFA:

See Annexed document.

L.S.K.

2. Backstopping officer:

The project had a difficult start due to the fact that no adequate counterpart personnel were available. However, due to the efforts of the National Project Director and the Director of CTA the situation changed promptly. The Scientific Advisor to the project was very helpful in putting the project on solid basis and gave a great deal of assistance in recruitment of the best available consultants in this field, and was very helpful in placement of trainees and in the study tours.

He also made it possible to get an excellent Chief Technical Advisor who helped in training and maturation of the team that can now function alone although it would have been desirable that he would have stayed another year. The project will be extended to the end of 1985 because a Phase II of the project is actually being prepared, the objectives being to assist in the applications of the Carbon Fibres and it is desirable that the Phase II will be a normal continuation of Phase I. On the whole the project has achieved its objectives and can be considered as a successful project.

G. What distribution of this PER is recommended?

The completed PER after endorsement will be sent to: CTA, SIDFA, DIO, PC/EVL, Registry

If you suggest additional distribution please indicate

☑ Resident Representative ☐ targeted beneficiaries

☐ UNDP Headquarters ☐ input for scheduled tripartite review or in-depth evaluation

☐ co-operating Government agency ☐ [illegible]

[illegible]

A. Causal Factors — Checklist

(Fill out and attach to PER)

		Not applicable	[illegible]			[illegible]
			Less than anticipated	As anticipated	Better than anticipated	
TECHNICAL ASPECTS						
1.	Personnel					
a)	Job descriptions appropriate to project/local needs			X		
b)	Experts' qualifications compared to job descriptions			X		
c)	Adequacy of international expertise to project/local needs			X		
d)	Ability of international staff to communicate knowledge to local staff			X		
e)	Assimilation of knowledge by national staff			X		
f)	Availability of technically qualified local personnel				X	
g)	UNIDO man-years available				X	
h)	Government man-years available				X	
	Other factors and/or comments:					
2.	Project Approach					
a)	Adequacy of project funding			X		
b)	Adequacy of government funding				X	
c)	Appropriate to project function (purpose)			X		
d)	Appropriate to local needs and demand			X		
e)	Appropriate to local cultural/technological conditions			X		
f)	Dissemination/multiplier effect to expertise provided				X	
g)	Complementary with other expertise existing/provided			X		
h)	Adequacy of national counterpart institution			X		
i)	Availability of reliable data/statistics		X			
	Other factors and/or comments:					
3.	Equipment					
a)	Quality of equipment procured with international resources			X		
b)	Local capability to maintain/repair project equipment		X			
c)	Quality of equipment procured/available from government resources			X		
d)	Adequacy of equipment to local technological needs			X		
e)	Timeliness of receipt of necessary equipment			X		
f)	Installation/use of equipment			X		
g)	Complementarity between equipment and training			X		
h)	Complementarity between equipment and expertise			X		
	Other factors and/or comments:					
4.	Training (on-the-job and fellowship)					
a)	Suitability of training to trainees' backgrounds			X		
b)	Suitability of training to trainees' assigned positions			X		
c)	Suitability of training to local needs				X	
d)	Suitability of training to local cultural/technological conditions			X		
e)	Length of training			X		
f)	[illegible] of training institution (fellowship training only)				X	
g)	Opportunity of trainees to apply new knowledge				X	
h)	Suitability of manuals/teaching materials					
i)	Complementarity between training and expertise			X		

A. Causal Factors — Checklist

(Fill out and attach to PER)

		Not applicable	Performance vs. Expectations: Less than anticipated	As anticipated	Better than anticipated	Check if important
j)	Turnover of trainees (on-the-job)			X		
k)	Turnover of fellowship recipients			X		
l)	Availability of qualified trainees/fellowship applicants			X		
m)	Return of fellowship recipients to project-related positions			X		
	Other factors and/or comments:					
	PERSONNEL					
1.	UNIDO/International Personnel					
a)	Awareness/agreement on project objective				X	
b)	Awareness/agreement on plans/approach			X		
c)	Adherence to work programme/schedules			X		
d)	Provision/continuity of project leadership				X	
e)	Willingness to work outside duty station				X	
	Other factors and/or comments:					
2.	Government Personnel					
a)	Awareness/agreement on project objective			X		
b)	Awareness/agreement on plans/approach			X		
c)	Adherence to work programme/schedules			X		
d)	Availability/continuity of project leadership				X	
e)	Willingness to work outside duty station				X	
f)	Adequacy of salaries and allowances			X		
g)	Qualified personnel remaining in project positions			X		
	Other factors and/or comments:					
	MANAGEMENT/ADMINISTRATIVE SUPPORT					
1.	UNDP					
a)	Support in preparing project document/revisions			X		
b)	Clarity/appropriateness of procedural guidelines			X		
c)	Timely provision of project funds			X		
d)	Response to requests for assistance			X		
e)	Guidance on local procedural formalities			X		
f)	Support in recruitment of local staff	X				
g)	Support in selection of local consultants	X				
h)	Support in obtaining supplies			X		
i)	Support in letting sub-contracts	X				
j)	Payment of salaries/allowances/expenses			X		
k)	Guidance on local political/institutional situation			X		
l)	Co-ordination with related projects			X		
m)	Assistance in [illegible] negotiations and contracts			X		
	Other factors and/or comments: UNDP played an important part in the success of the CTA/UNIDO/UNDP sponsored conference					

A. Causal Factors — Checklist

(Fill out and attach to PER)

	Not applicable	Performance vs Expectations: Less than anticipated	As anticipated	Better than anticipated	Check if important
2. UNIDO Headquarters					
a) Support in preparing technical parts of draft project documents				X	
b) Support in developing technical approach			X	X	
c) Overall adequacy of technical backstopping					
d) Support in preparing project documents/revisions				X	
e) Guidance/assistance on design/evaluation methodology					
f) Clarity/appropriateness of procedural guidelines				X	
g) Timely provision of project funds				X	
h) Response to requests for assistance				X	
i) Support in obtaining supplies				X	
j) Support in letting sub-contracts	X				
k) Payment of salaries/allowances/expenses			X		
l) Guidance on local political/institutional situation			X		
m) Co-ordination with related projects				X	
n) Timely recruitment of international staff			X		
o) Timely procurement of equipment			X		
p) Timely placement of fellows			X		
q) Timely reply to communications			X		
r) Usefulness of replies to queries			X		
s) Guidance on policies and procedures					
t) Clear assignment of project authority/responsibility				X	

Other factors and/or comments:

UNIDO office provided excellent support in conducting CTA/UNIDO/UNFSSTD sponsored conference at CTA.
Wherever UNIDO representatives were in the Project site, a moral support was given especially to the Group working and this Project. A sign of encouragement and appreciation was given. This is very necessary in the development of this type of technologies.

	Not applicable	Less than anticipated	As anticipated	Better than anticipated	Check if important
3. Senior Industrial Development Field Adviser's Office					
a) Support in preparing technical parts of draft project documents					
b) Support in establishing and defining objectives			X		
c) Support in developing technical approach				X	
d) Guidance on project background			X	X	
e) Guidance on national political/institutional situation			X		
f) Guidance on UNIDO procedures and policies			X		
g) Guidance/assistance on local administrative formalities			X		
h) Guidance/assistance on design/evaluation methodology			X		
i) Promotion/co-ordination of local contacts				X	
j) Replies to queries				X	
k) Support in recruitment of local staff	X				
l) Support in selection of local consultants	X				
m) Support in obtaining supplies			X		
n) Support in letting sub-contracts	X				
o) Secretarial help/support			X		
p) Payment of salaries/allowances/expenses			X		
q) Co-ordination with related projects				X	
r) Assistance from HQ				X	

Other factors and/or comments:

SIDFA office also has played an important part in the success of CTA/UNIDO/UNFSSTD conference conducted at CTA.

A. Causal Factors — Checklist
(Fill out and attach to PER)

		Not applicable	Performance vs Expectations: Less than anticipated	As anticipated	Better than anticipated	Check if significant
4.	**Government**					
a)	**Provision of physical facilities and equipment**				X	X
b)	Availability of secretarial help			X		
c)	Provision of office material/equipment			X		
d)	Provision of transport facilities			X		
e)	Provision of storage facilities			X		
f)	Provision of counterpart staff			X		
g)	Provision of funds			X		
h)	Guidance on administrative formalities			X		
i)	Support in undertaking administrative formalities				X	
j)	Maintenance and repair of equipment			X		
k)	Availability of policy and management officials			X		
	Other factors and/or comments: The counterpart coordinator T.Cel Kessel has taken special interest in the success of conference. At all stages, he has been confiding with CIA in the general management and methodology in the development of this technology. His active participation bousted the confidence in the group personnel and encouraged them in active participation in the developmental activities.					
5.	Other Participating Agencies					
a)	Timely recruitment of staff	X				
b)	Timely purchase/provision of equipment	X				
c)	Timely placement of fellows	X				
d)	Timely replies to administrative queries	X				
e)	Usefulness of replies to queries	X				
	Other factors and/or comments:					
	EXTERNAL FACTORS					
a)	Government receptiveness to change			X		
b)	Continuity in economic/social/industrial policy			X		
c)	Enforcement of relevant legislation	X				
d)	Support by industry organizations				X	
e)	Support by other organizations (specify)	X				
f)	Co-ordination among Government agencies	X				
	Other factors and/or comments:					

PROGRAMA DAS NAÇÕES UNIDAS PARA O DESENVOLVIMENTO NO BRASIL

UNITED NATIONS DEVELOPMENT PROGRAMME IN BRAZIL

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REF.:
FILE:

Brasília August 7th, 1984.

COMMENT FOR ENDORSEMENT OF PER COVERING PERIOD DEC 2nd 83 - JUNE 29th 84 OF BRA/81/T01 -(PAG 5)

Enclosed is herewith PER with the following comments (1):

a) Starting January 1984, Brig. Piva took office as Director of "Centro Tecnológico Aeroespacial" (CTA). He is a PhD from Massachussets Institute of Technology (MIT) and as such, he is really able to understand R and D work. I have talked with him after his becoming the Director. He fully supports the project and is quite conscious that it constitutes a very important venture in terms of product to be produced (carbon fiber) and in terms of creating a team of innovative people with autonomy to keep on working with this or other fibers and materials in the future. (2) He is quite aware of the need to keep on adding highly qualified personnel to the team, to keep the present personnel in the project and also to continue improving it. He is also conscious about the leadership problem, the need to complete the physical facilities and about the language problem.

b) The problem of leadership, which I discussed in the previous PER and letters, is in the way of being solved via the permanence of Dr Nagabushanam at CTA. In this aspect, Brig Piva and Cnel Kessel are undertaking all efforts to make this possible. I want to insist that we should also do our utmost to help in this process(3). I think the presence of Dr Nagabushanam, using him as master, is absolutely essential to form a national leader.

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(1) The comments partly refer to my previous comments to last PER, please refer to them.

(2) By this I do not mean that we did not have such a strong backing from Brig. Menezes, who was an extremely able man too and who gave full support to our project.

(3) I think we will have to keep investing in sending him to congresses (CONT).

c) The process of maturation of the team continues on going, the processes and technologies to produce the carbon fiber and precursor at bench scale and pilot plant are being developed. The necessary equipment is being put into place and in operation. Supposing that we successfully complete all that process, the introduction of all that at the industrial scale will become important. In this sence, the CTA is taking the right steps too, namely:

   i- the contract between CTA and COPENE which would include the financial help from the latter in the final stages of development of the technology at the CTA and the responsability of COPENE for the work which is being carried out at FISIBA (4) COPENE will inject the necessary money to complete the work at pilot scale there and to install the subsequent industrial production.

   ii- Cel Kessel is already negotiating with a company, the technology for the production of oxidized PAN (at rate of 5 ton/month), which is very important in terms of stabilizing part of the production cycle.

   iii- Mitzui has been initiating the production of prepeg using imported carbon fiber. This is very important, since they will open the market for carbon fiber (at EMBRAER and others). Subsequently, once the local production of carbon fiber attains the right quality, it will be only a matter of substituting the imported carbon fiber by the national one.

---

(3) (CONT.) outside Brazil in such a way that he does not feel that he is getting out of the "jet set" in his area of expertise.

(4) FISIBA is a comparatively quite small company when compared with the mother company, COPENE, besides that, FISIBA is going through a bad financial situation.

d) At CTA, Dr Nagabushanam continues to be an extremely important part in the continuing maturation of the team. His presence is essential in the completion of the whole process in which we are involved.

e) In terms of actions required , I would add:

i- To complete as soon as possible the process (be it with the Sta Clara company in Brazil or any other) to make the production possible and put into operation the stabilization furnace at the appropriate capacity (1 - 3 Tons/year). Unit reponsable: CTA with UNIDO's help.

ii- Even if it may appear a sideline matter, the team has to become more proficient in English as to extract the most out of the consultants and scientific and technical literature in general.

5.12.18
AD.87.04