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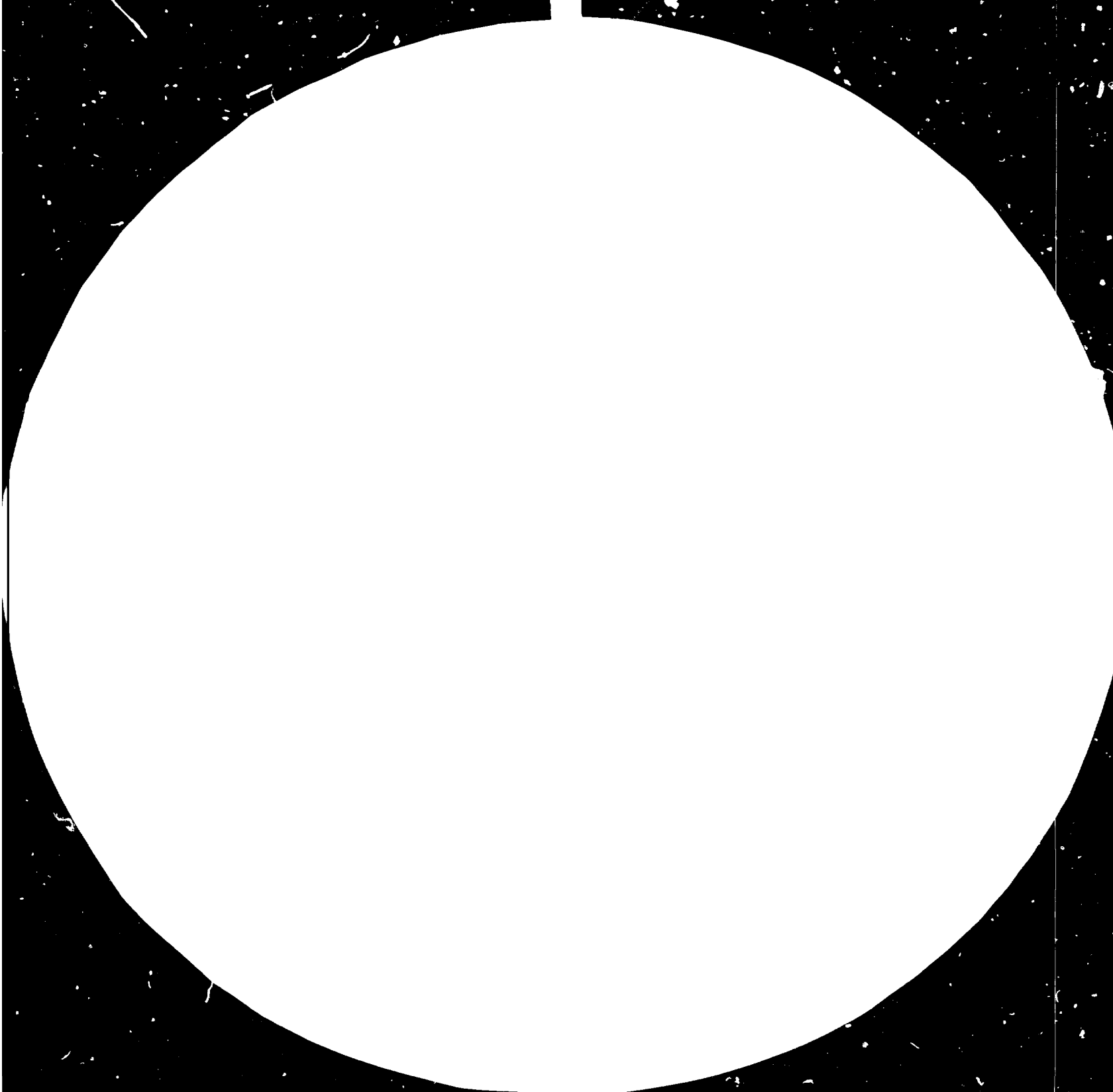

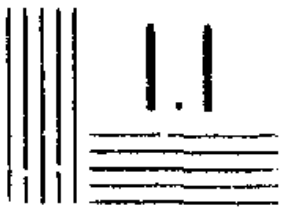

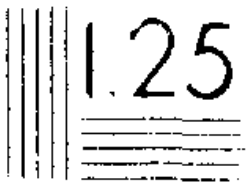

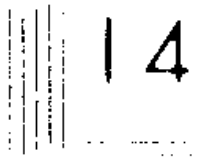

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1983

Report of my activities as UNIDO-consultant in the

carbon fibre project in Sao Jose dos Campos, Brazil

ST/ECLA/81/001/11-63/32.1.A

Duration: Two months; from 16. February to 15. April

Situation of the project when I arrived

After my arrival in São José dos Campos I was informed about the state of the project by Dr. Nachtigall and Eng. Simionato.

- oxidation experiments:
  Following the results of my previous stay and the advice of Dr. Jacobsen and Dr. Kalnin, two-step oxidation experiments with varied amount of forced air have been performed. The Courtaulds-PAN, prestretched up to 15% at 190°C was used for all experiments.

  Basing on the expected results of these experiments, a paper for the Carbon Conference in July 1983 in San Diego, Cal. was announced. On my arrival, first results from these experiments were ready.

- analytical methods
  I knew already by private communication with the Brazilian staff, that Eng. Arnaldo, who was trained by me last year in DT- and IR-analysis of PAN, left the group.
  The transfer of his knowledge to the other members of the group was limited, only some basic experiments were done in the meantime.
  On my arrival, the DTA-equipment was in São Paulo for repair.
  IR-analyses are now done by a student, who is working beside his studies in the group.
  The sink-float method for the measurement of fibre densities, also introduced by me last year, is now a standard method and extensively used.

Activities

The subjects of my activities were more or less the same like during my stay in 1982:

- Planning of oxidation experiments as well as the discussion of the results (with respect to the announced paper for the Carbon Conference in San Diego).
- Analytical methods for the characterization of initial PAN, oxidized and carbonized fibres (re-introduction and deepening respectively).
- Planning basic research programs, which help for a better understanding of the changes of PAN during the processing into C-fibres.

Oxidation experiments

Initiated by the advice of Dr. Jacobsen and Dr. Kalnin, oxidation experiments with Courtaulds-fibres had been performed. The fibres had been prestretched at 190°C in air to 0, 5, 10 and 15% respectively. The resulting fibres were oxidized at constant length in two steps (step 1: 230°C, 40 minutes; step 2: 265°C, 60 minutes). The only varied parameter during these steps was the amount of forced air in the furnace (0, 15, 35 l/min). The oxidized fibres were carbonized in argon up to 1100°C allowing at about 7% shrinkage. Oxidized and carbonized fibres were characterized with respect to density and mechanical properties. These results have been ready just on my arrival and were used as a base for the extended abstractof the San Diego-paper (enclosed to this report) and for the planning of further experimental work for the paper. The main aspect of this work was the optimization of the residence time at the oxidation steps with respect to the maximum tensile strength of the resulting C-fibres. An amount of 15 l/min forced air during oxidation was used for these experiments. It was found, that a residence time of 40 to 60 minutes is optimum for each oxidation step. Prestretching to 5-10% resulted in maximum tensile strength and density values of C-fibres

Because a shortening of the oxidation time is expected by the variation of the amount of forced air (low amount at 250°C, high amount at 265°C, see extended abstract), such experiments are planned for the time after my leave.

Analytical methods (DTA, TMA)

Because of the limited transfer of knowledge from Eng. Arnaldo to the other members of the carbon fibre group I had to introduce again procedures and interpretation possibilities for DT-analyses nearly completely.
As Eng. da Silva is now responsible for the DTA-work at the group, I teached him extensively. Because of a delay in the repair of the DTA-equipment, it was only ready during the last three weeks of my stay. The time before was used to discuss the Differential Thermal Analysis and its suitability in the carbon fibre work theoretically.
I worked out instructions for the use of the DT-equipment and for qualitative and quantitative interpretation of data from DT-experiments. The evaluation of heat capacities of fibres, of heat enthalpies, the interpretation of the cyclization peak of PAN as well as the calculation of kinetic data for the cyclization are described in these instructions.
All techniques were trained experimentally, when the equipment was back from the repair.

I also introduced the work with the Thermo Mechanical Analyzer (TMA), which is an accessory of the DTA-equipment. The TMA is capable to measure the shrinkage of PAN-fibres during cyclization. I instructed the group (mainly Eng. da Silva) in the techniques and the qualitative and quantitative interpretation of TMA-data.

There was no time to do systematic work with DTA and TMA. Therefore I worked out a program for systematic studies of the cyclization by DTA and TMA in combination with IR-studies. A better understanding of the chemical changes of PAN during theoxidation should result. If the experiments are done carefully, the results can also be a base for further publications.

Comments and recommendations

Comparing the work of the carbon fibre group during my stay in 1982 and this time, I had the impression, that the experimental work is now done more systematically and with a better understanding of the process. This may be due to the advices of the UNIDO-experts Dr. Jacobsen and Dr. Kalnin, but also to the supervision by Dr. Nagabhushanam, who takes care on the careful performance of the programs initiated by the experts.

The announcement of the paper for the Carbon Conference had a strongly positive effect on the motivation of the Brazilian staff. I planned the DTA- and TMA-programs in a manner, that publications from the results should be possible. By that, the Brazilians should be motivated also in these basic studies. Dr. Nagabhushanam will also take care for their performance. I will keep contact to the group also in the future in order to help if necessary and possible, especially for the completion of the paper for San Diego.

Although the knowledge of the Brazilian group is better than last year, it is necessary to improve more the understanding of the chemical and physical changes of PAN during oxidation and carbonization, if the development of a Brazilian precursor fibre shall be successful.