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Main Vacuum Chamber NI/GP/60/20

Under Item 2. The total commitment on the Marston Contract is stated to be £175,000 up to 31.3.60. This is incorrect and this sum is the Marston expenditure against this contract at that date. The Authority commitment at 31.3.60. is estimated to be £219,000 plus any cancellation charges. The Authority commitment is now estimated to have reached the ceiling on the contract, i.e. £230,000, hence the urgent need to increase the limit of expenditure.

F.M. TELLING

11.5.60.

4th May, 1960

NI.(GP)(60)20
(PSPC/P65)

NATIONAL INSTITUTE FOR RESEARCH IN NUCLEAR SCIENCE

GENERAL PURPOSES COMMITTEE

THE MAIN VACUUM CHAMBER FOR NIMROD

Note by the Deputy Chairman of the Project Committee

SUMMARY

This paper requests approval for the authorised limit of expenditure of the development contract with Marston Excelsior Ltd. to be increased from £230,000 to £398,000. This increase can be met without over-committing the present approved estimate for the Vacuum System.

1. INTRODUCTION

1.1 In February 1959 a contract for the development and supply of the main vacuum chamber was placed with Marston Excelsior Ltd. (Paper No. NI.GP.59.1 refers).

1.2 The development section of this contract required Marston Excelsior Ltd. to undertake a technical study to determine suitable constructional techniques, the production of models and samples as necessary to establish techniques, and the manufacture of one complete octant of the main vacuum chamber.

1.3 At the time of placing the contract the development stage had a limit of expenditure of £100,000. In November 1959 this limit of expenditure was increased to £230,000. (Paper NI.GP.59.44 refers).

2. ESTIMATED COSTS

The cost of the development stage of the contract has been re-examined on the basis of present knowledge and the new estimate is £398,000. The break-down of the original and revised estimate is as follows:-

	<u>Original</u> Jan.1959 £	<u>Revised</u> Oct. 59 £	<u>Revised</u> Mar. 60 £
Technical study and development	30,000	55,000	160,000
Plant and tools	60,000	150,000	201,000
Manufacture of one complete octant	25,000	25,000	37,000
	<u>£115,000</u>	<u>£230,000</u>	<u>£398,000</u>

Total commitment to 31st March 1960 = £175,000.

3. TECHNICAL STUDY AND DEVELOPMENT

3.1 The contract called for the first section of the outer chamber to be delivered by 31.10.59. The original estimate of £30,000 for technical study and development was based on a period to meet this delivery date. In October 1959 the estimate was revised to £55,000 since it became apparent that appreciably more development work than was first envisaged would be necessary to produce the sections. The development stage however again became protracted due to difficulties in establishing basic processes of a material for which no previous manufacturing experience was available. This has resulted in an inevitable rise in the development costs now estimated to be £160,000.

3.2 One of the major difficulties in the development programme has been to establish a resin system and curing cycle which gave a laminate compatible with the manufacturing processes and physical requirements. The amount of research necessary to establish this fundamental process is summarised in chronological order in Appendix I to this paper. After intensive experimental work encouraging results have been achieved using a new accelerator which not only gives a laminate superior to that previously developed, but also resolves the difficulties experienced in jointing the material. Subject to certain fatigue tests and satisfactory irradiation results the resin formulation using the new accelerator will be adopted for production requirements. This will minimise the use of refrigerant in the manufacturing process.

3.3 A great deal of development work has been necessary to establish a technique of applying a metal lining to the inner and header sections, and tests are continuing on samples using stainless steel and aluminium foils bonded to epoxide laminates.

3.4 The development of pilot plant tools has been necessary to establish fabrication methods and also prepare laboratory samples for testing purposes. The cost of raw materials, i.e. glass cloth, resin and hardeners, is high averaging about 7/6d. per lb. The cost of producing small samples for laboratory testing is estimated at £3 per lb. weight.

3.5 The cost of designing the complex production plant and tooling has also been included in the development charges at an estimated cost of £40,000.

3.6 The training of labour for the production and inspection process is another item charged to the development contract and the estimated costs in this respect are £24,000.

4. PLANT AND TOOLS

The cost of providing special plant for the production of the outer, inner and header sections is estimated to cost £144,750. In addition, general plant is estimated to cost £56,200. A complete list of this plant is shown in Appendix II.

5. MANUFACTURE OF ONE PROTOTYPE OCTANT

Manufacture of the first outer section is expected to commence during early May 1960 with completion during June 1960. The close proximity of the production processes together with the knowledge gained from the development stage has enabled a revised estimate to be made of the cost of the prototype octant. The previously suggested figure of £25,000 is now known to be too low and expenditure in this respect is estimated to be £37,000.

6. COMMITMENTS

6.1 The present Treasury approval for the Vacuum System is £780,000, of which the commitments are approximately £420,000.

6.2 Approval by the General Purposes Committee to increase the vacuum chamber development contract by a further £168,000 would therefore increase the present commitments to £588,000 which is still within the Treasury approved estimate.

7. RECOMMENDATIONS

7.1 Paper NI.GP.60.19 on the NIMROD machine estimates takes into account the revised estimate for the main vacuum chamber, but authority is requested to increase the authorised limit of expenditure of the contract with Marston Excelsior to £398,000 prior to Treasury consideration of the revised NIMROD estimates, sufficient funds being available in the present approved estimates to prevent over-commitment.

7.2 It is therefore recommended that approval be given to the following estimated costs:-

Development of a prototype octant	£160,000	280,000
Manufacture of a prototype octant	£ 37,000	63,000
Plant and tools for the manufacture of a prototype and eight production octants	£201,000	360,000
	<u>£398,000</u>	<u>£703,000</u>

F. A. VICK

Deputy-Chairman, NIMROD Project Committee

NIMROD PROJECT

Summary of the development work carried out by Marston Excelsior Ltd., to
select a suitable resin system and curing cycle for the main vacuum chamber

- Jan. 1959 Programme prepared to select suitable resin system and cure.
- 6.4.59. For the outer vessel work should concentrate upon evolving a suitable wet lay up formulation.
- Marston favoured the use of the MNA system to the DDM system. A graph of cure time against shelf life of the wet ends was tabled demonstrating that T.D.M.P. was the preferred accelerator.
- The recommended minimum cure was 4 hrs. at 150°C. The recommended optimum cure 6-8 hrs. at 180°C. This was still under investigation.
- 27.4.59. At a meeting at A.E.R.E. it was decided to use the F/MNA system.
- 29.4.59. T.D.M.P. accelerator unstable in the presence of MNA. Therefore B.D.M.A. to be used. Cure at 6 hrs. at 200°C. - laminate of high strength with opacity. Cure at 130°C. - laminate of low strength, a compromise temperature is being sought. Tools are being designed to carry out Post Cure at 150°C. with a 7 hr. curing cycle.
- 4.5.59. The choice of accelerator undecided because recent experience had shown separation of crystalline material on storage.
- Decision on curing and post curing times and temperature not so straightforward as at first supposed. A sample laminate following a severe post curing had developed whitening. (Opaque sample). The temperature of curing, the time at temperature, and the time to reach temperature, as well as the post curing effect of radiation were all factors that must be considered.
- 20.5.59. Standard Resin formulation to be MNA system without diluent.
- Separation of crystalline material. Crystals were methyl ('Nadic') acid and they were not harmful at the concentration present.
- A.E.R.E. tabled samples of a layer of single glass cloth impregnated with resin and initially cured at different temperatures before being post cured.
- Samples cured at:-
- (a) 100°C. were clear and water white with good surface finish and no blisters.
 - (b) 150°C. were clear and golden with a poor finish and blisters.
- Strain spots through the polarising microscope were:-
- (i) A maximum for samples cured at 150°C.
 - (ii) Less noticeable for the layer cured at 200°C.
 - (iii) Least noticeable for the layer cured at 100°C. and post cured at 200°C.
- The opaque sample (150°C. initial cure plus 180°C. post cure) reported above, on pumping trials leaked through one fissure. It showed intense cracking when viewed by transmitted light using a stereoscopic microscope.

Evidence pointed towards a low initial cure followed
by subsequent post cure

Mark I recipe.

27.5.59. The recipe optimum proportions are as follows:-

Resin 100 parts Araldite 999A (F to a tighter viscosity range).
Hardener 80 parts MNA (Methyl Nadic Anhydride).
Accelerator 0.3% BDMA (Benzyl Dimethylamine). Can be increased
to 3% for faster cure for dorsal splicing where wet
ends no longer required.

The Mark I recipe plus reactive diluent (Butyl glycidyl ether 556A)
regarded as Mark II recipe.

Laboratory made samples had lower strength than those prepared in
the matched dies from the shop floor.

Results at A.E.R.E. show that there is less strain and fewer resin cracks
in samples cured at moderate temperatures, prior to post curing.

1.6.59. A.E.R.E. experiments suggest that the use of low temperature was likely
to be beneficial. Photographs showing strain patterns in case resin
samples show that strain was less after a low temperature preliminary
cure, followed by normal post curing. Examination of single layers of
impregnated glass cloth under a polarising microscope demonstrated that
strain, cracking and shrinkage were less at low curing temperatures.

A.E.R.E. tabled samples of single layer glass/resin castings prepared
at different temperatures, from resin which had been subjected to
vacuum at 90°C. These samples cured at high temperatures showed
fewer voids. It was agreed this merited further investigation.

24.6.59. Subjecting the resin to vacuum has the following advantages:-

Reduces the danger of void formation at high temperature.
Prevents the formulation of the crystal impurities.
Facilitates the wetting out of the cloth.

Laminates cured for 5 hrs. at 150°C. and those cured with an
initial period at a lower temperature, the overall time remaining
the same, showed little difference in strength. This investigation
to be extended.

29.6.59. It was agreed to prepare a formal document stating the materials
etc. to be used for the outer vessel.

Early work had shown that a 5 hr. flat curing cycle at 150°C. had
given the highest strength.

A higher strength still could be obtained by a slow period of
heating to this temperature.

Periods of heating at both 90°C. and 115°C. before rising to 150°C.
for the main cure, showed that 115°C. was detrimental, and cure at
90°C. did not show any advantage.

Long heating at 150°C. caused loss of strength, presumably through
degradation.

The recommended cure cycle was therefore:-

1 hr. heating to 150°C.
2 hr. at 150°C.

4.8.59. A.E.R.E. Tests on Mk. I recipe

Resin samples cured at 100°C, 120°C, 140°C, 160°C, 180°C and 200°C
for 1 hr. and a second set of samples cured as above and each post
cured for 4 hrs. at 150°C.

On examination, using a strain viewer, uncured samples cured
at 100°C. and 120°C. showed very little strain. Those cured at
higher temperatures showed strain to an increasing degree. Samples
cured at 180°C and 200°C. being severely strained.

APPENDIX I (contd.)

For the post cured samples again evidence of strain increased with temperature, but each casting in general showed less strain than the corresponding sample "unpost-cured."

Report P9527/1 and P9527/2 received from Marston.

Recommended	Resin	Araldite F
	Hardener	M.N.A.
	Accelerator	(BDMA 0.5%
		(TDMP 0.5%
		(K61B 0.8%

Minimum Cure 4 hrs. at 150°C.
Optimum Cure 6-7 hrs. at 180°C.

10.8.59.

MKI Recipe proposed cure schedule

- (1) Place male tools in position.
- (2) Start to heat. (3) At 50°C. put pressure on.
- (4) Heat dies and punch from room temp. to 90°C. $\pm 5^\circ\text{C}$. in $\frac{1}{2}$ hr.
- (5) Stabilise both die and punch at 90°C $\pm 5^\circ\text{C}$. as quickly as possible.
- (6) Heat both punch and die from 90°C. to 145°C. $\pm 5^\circ\text{C}$. in $\frac{1}{2}$ hr. keeping the temperatures of the die and punch in step $\pm 5^\circ\text{C}$.
- (7) Hold at 145°C. $\pm 5^\circ\text{C}$. for 2 hrs.
- (8) Cool in not less than 1 hr.

MK II recipe (MK I recipe with diluent). Optimum curing schedule time twice as long.

30.10.59.

MNA ordered for Outer Inner and Header vessels.

9.11.59.

Wet ends have been stored at -20°C. for 10 weeks, and when spliced are satisfactory.

A.E.R.E. reported that heat distortion temperature of laminates to the preferred recipe dangerously low. Flexural strength fell off markedly with increase in temperature.

Flexural strength tests carried out at an elevated temperature on shop made laminates was not nearly so severe as laboratory samples led to believe.

Issue 2 of Specification in draft form:-

Epoxy Resin	100 $\pm$ 0.1 parts by weight
M.N.A.	80 $\pm$ 0.1 " " "
BDMA	0.3 $\pm$ 0.01 " " "

Cure

- (1) Place male tools at 40°C. in position.
- (2) Start to heat.
- (3) Heat dies and punch from 40°C. to 90°C. $\pm 5^\circ\text{C}$ in not less than 15 minutes.
- (4) Stabilise both die and punch at 90°C. $\pm 5^\circ\text{C}$. for 30 mins.
- (5) Heat both punch and die from 90°C to 145°C. $\pm 5^\circ\text{C}$. in not less than 15 mins., keeping the temperature of the die and punch in step $\pm 5^\circ\text{C}$.
- (6) Hold at 145°C $\pm 5^\circ\text{C}$ - 2 hrs.
- (7) Cool in not less than 2 hrs.

7.12.59.

A.E.R.E. strongly suggest that the curing temperature be increased from previously accepted 2 hrs. to a period up to 12 hrs.

A.E.R.E. consider that $\pm 10\%$ variation from the stoichiometric mix appreciably affect heat distortion point and flexural strength at elevated temperatures.

A.E.R.E. expressed anxiety that shrinkage of the laminate with age might occur through undercure.

Lateral shrinkage of laminates during post cure:-

0.2% after 3 hrs. at 150°C. No further shrinkage up to 20 hrs. at 150°C.

Lateral shrinkage after 10^8 rads. irradiation 0.1%.

No shrinkage after similar irradiation on post cured sample.

A.E.R.E. pointed out that originally a curing cycle of 5 hrs. had been used but this had been changed to 2 hrs.

Marston explained that the change from 5 hrs. cure to 2 hrs. had been introduced deliberately following tests on strength at room temperature.

Preliminary results of shrinkage tests carried out at Marston on shop made laminates showed that a lateral shrinkage of 0.04% was occurring.

Factors affecting Cure

1. Accelerator content. Could not be increased - wet end life already too short.
2. Temperature of Cure. A raised temperature of cure would mean delays whilst further experimental work was conducted at the higher temperature.
3. Time of cure. Increase of curing time,
 - (a) Would probably increase the life required of wet ends.
 - (b) Would probably increase the number of punches required.
 - (c) May necessitate a reconsideration of the electrical supply to each moulding table.
 - (d) Would necessitate careful consideration of the tooling expansion problems.

In the present 3 punch system, each laminate section was at a temperature above ambient for no less than 6 hrs., i.e.

- 1 hr. heating
- 2 hrs. at temperature
- 3 hrs. cooling.

A.E.R.E. suggested that if a 5 hr. cure is required this might be achieved with 3 punches by extending the time at temperature to 3 hrs., or holding the "cooling" tool at temperature for the maximum possible time.

A.E.R.E. also pointed out that 10°C rise in curing temperature was equivalent to doubling the time of cure.

7.1.60.

Marston reported the following strength figures of laboratory made laminates to MK I formulation and cure.

Flexural strength 57,000 p.s.i.
 Strength retention at 35°C. 89% min.
 " " " 50°C. 75% min.
 Irradiation 5.6 x 10⁸ rads. - no change.
 Fatigue (10⁷ cycles) 4 tons/sq. in.

A.E.R.E. reported that shop made laminates irradiated up to 5 x 10⁸ rads. had shown a growth which seemed to be related to the degree of radiation. Post cured samples showed an inconsistent scatter in dimensional changes and subsequent irradiation gave a fairly constant growth which was not related to degree of cure. The maximum growth in length had been 0.1%

Marston reported that laboratory tests on a single shop made moulding made at 155°C. had shown no marked improvement as judged by strength at temperature or sensitivity to acetone. It might be possible to increase slightly the time at the curing temperature and adjust the curing time but the 6 hr. punch cycle must be retained.

At a meeting at A.E.R.E. it was decided in view of the doubts raised that the temperature of cure should be raised to 155°C.

Feb. 1960. Some discrepancies had been found at Marston between laboratory sample and shop made laminates, further tests are in hand.

Work is proceeding to investigate:-

- (i) The method of mixing the constituents of the resin.
- (ii) The effect of vacuum treatment of the resin, as this could be responsible for removing accelerator or stray inhibitors.
- (iii) The effect of delay in using the resin after mixing.

The possible effect of these variables will be investigated on gel time and on finished laminates.

Glass transition point (0.3% BDMA, MNA system)

<u>Cure</u>	<u>Transition point</u>
2 hrs. at 145°C.	19°C.
5 hrs. at 145°C.	27°C.
10 hrs. at 145°C.	35°C.

7.3.60.

Work in progress:-

- (i) To determine the compression creep of laminates which have received different degrees of cure.
- (ii) To correlate this creep with a reliable method of determining the degree of resin cure.
- (iii) To ascertain the effect of different curing times on the second order transition temperature of the resin.
- (iv) To investigate the connection between the compression creep of a laminate at 35°C. and the second order transition temperature of the resin used.

Indications showed that laminates based upon CIBA accelerator 33/1266 (as a replacement for BDMA) were very favourable if judged on the basis of strength at elevated temperatures. Work on this new accelerator would continue in parallel with the BDMA recipe and similar tests to those being carried out on the standard recipe would need to be applied to the new formulation.

4.4.60.

Preliminary work using the new accelerator suggests that in addition to doubling the life of wet ends, use of a concentration on 0.6 parts would in 2 hours at temperature, give a degree of cure comparable with 10 hours using BDMA. Preliminary fatigue tests at room temperature are being arranged and equipment to measure fatigue at elevated temperatures is being obtained. Subject to suitable irradiation tests and no unforeseen difficulties on the full scale tools the following is suggested for production processes:-

Formulation:- 100 parts Araldite 999A
80 parts M.N.A.
0.6 parts 33/1266

Cure:- Heat to 130°C. in $\frac{1}{2}$ hour.
Hold at 130°C. for 1 hour to gel resin.
Raise temperature to 155°C. in $\frac{1}{4}$ hour
for $1\frac{1}{2}$ hour.
Cool in 3 hours.

NIMROD PROJECT

Plant and Tools for the Main Vacuum Chamber

1. GENERAL PLANT

Checking Plate	5,950
5 ton Crane	3,500
Extensions to 30 cwt. Gantry	600
Transport Frame	1,600
Lifting Frames	4,500
Truck	200
Checking and Drilling Jigs	10,000
Metrology	3,000
Fume Extraction	3,000
Portable Drill	350
Cloth Prewetting Machine	1,000
Sundry Small Tools	1,000
Inspection Equipment	4,000
Transport Frames	7,500
Pilot Plant Work	10,000
Total Cost of General Plant	<u>£56,200</u>

2. SPECIAL PLANT FOR MANUFACTURE OF OUTER COMPONENT

A. <u>Side Moulding Plant</u>	
Matched Dies, 15 Bottom, 6 top	13,500
Extra Punch	790
Matched Dies, 6 special top and bottom tools	7,000
Bed Plate Moulding Section	3,520
Bed Plate Light Jig Section	1,500
Production Press	1,600
Lifting gantry and rails	600
Heating elements	1,340
Controllers and Contactors	1,650
Special grillage for centre section	270
Erection	5,500
	<u>£37,270</u>

SPECIAL PLANT FOR MANUFACTURE OF OUTER COMPONENT (continued)B. Dorsal Splicing Plant

19 Bottom Dies	4,600
3 Top Dies	1,000
Supporting framework and Clamps	6,000
Heating Elements	600
Lifting gantry	600
Controllers and Contactors	750
Erection	2,000
	<u>£15,550</u>

C. Special End Flange Tools£6,0003. SPECIAL PLANT FOR MANUFACTURE OF INNER COMPONENTA. Side Moulding Plant

Matched Dies, 14 bottom, 6 top	13,500
Extra Punch	800
Matched Dies, 6 special top and bottom tools	6,000
Bed Plate Moulding Section	3,500
Bed Plate Light Jig Section	1,355
Production Press	1,600
Lifting gantry and rails	600
Heating Elements	1,300
Controllers and Contactors	1,450
Special grillage for centre section	275
Erection	4,000
	<u>£34,380</u>

B. Dorsal Splicing Plant

19 Bottom Dies	3,000
3 Top Dies	750
Supporting framework and Clamps	5,000
Heating Elements	500
Lifting gantry	500
Controllers and Contactors	750
Erection	2,000
	<u>£12,500</u>

C. Special End Flange Tools£6,000

APPENDIX II (contd.)

4. SPECIAL PLANT FOR MANUFACTURE
OF HEADER COMPONENT

A.	Side Moulding Plant	10,000
	Special Side Moulding Plant	3,000
	Bed Plate (Splicing Section)	1,000
	Bed Plate Jig Section	3,000
	Press	-
	Gantry	500
	Heating Elements	1,000
	Temperature Controllers	700
	Floor Steelwork	-
	Erection	2,000
		<u>£21,200</u>
B.	<u>Dorsal Splicing Plant</u>	
	Dies	1,000
	Punches	500
	Supporting framework	3,000
	Heating Elements	1,000
	Gantry	600
	Temperature Controllers	750
	Erection	1,000
		<u>£7,850</u>
C.	End Flange Tools	<u>£4,000</u>

SUMMARY

1.	General Plant	56,200
2.	Special Plant for Outer Component	58,820
3.	Special Plant for Inner Component	52,880
4.	Special Plant for Header Component	33,050
		<u>£200,950</u>